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# CHEMICAL ENGINEERING

## VOLUME TWO

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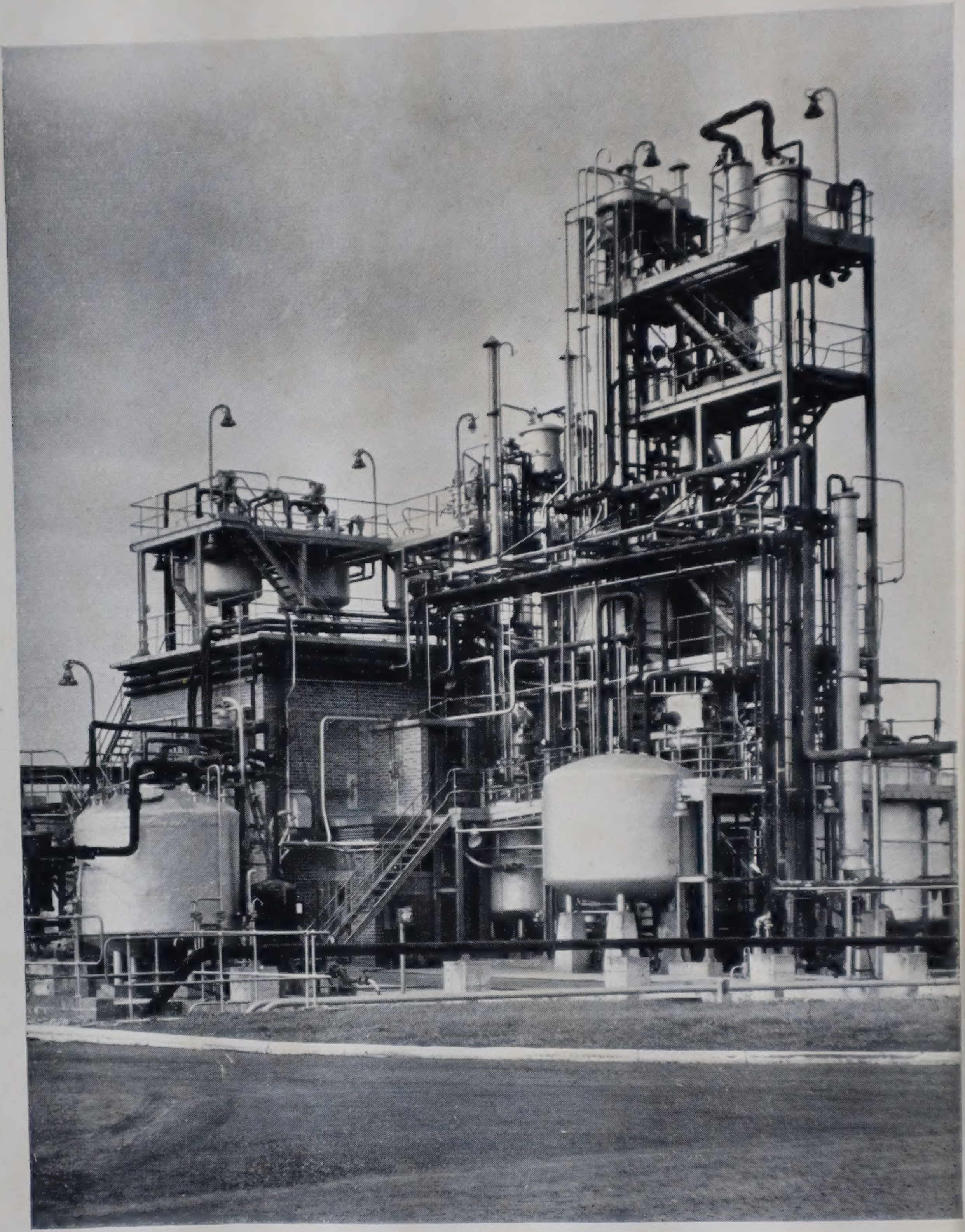

Plant for the manufacture of synthetic detergents

*Frontispiece*

# CHEMICAL ENGINEERING

## VOLUME TWO

## UNIT OPERATIONS

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*Note to the Second Impression*

No major revisions have been made in the Second Impression, but the opportunity has been taken to make a number of small corrections. We should like to thank those readers who have pointed out errors and shall be pleased to know if further shortcomings are found.

# PREFACE

In presenting Volume 2 of Chemical Engineering, it has been our intention to cover what we believe to be the more important unit operations used in the chemical and process industries. These unit operations, which are mainly physical in nature, have been classified, as far as possible, according to the underlying mechanism of the transfer operation. In only a few cases is it possible to give design procedures when a chemical reaction takes place in addition to a physical process. This difficulty arises from the fact that, when we try to design such units as absorption towers in which there is a chemical reaction, we are not yet in a position to offer a thoroughly rigorous method of solution. We have not given an account of the transportation of materials in such equipment as belt conveyors or bucket elevators, which we feel lie more distinctly in the field of mechanical engineering. It will be noticed that nothing has been said of automatic control. It is our opinion that this very important feature in the design of chemical plant is best illustrated by considering actual practical problems, and it is not appropriate to do so in a work of this nature. In any case, the subject of automatic control has become so complicated and extensive that it is demanding a treatment by itself. The omission of a chapter discussing the design of reactors arises from the fact that this subject also requires extensive consideration, and a specialist work, such as *Chemical Process Principles* by HOUGEN and WATSON, should be consulted.

In presenting a good deal of information in this book, we have been much indebted to facilities made available to us by Professor NEWITT, in whose department we have been working for many years. The reader will find a number of gaps, and a number of principles which are as yet not thoroughly developed. Chemical engineering is a field in which there is still much research to be done, and, if this work will in any way stimulate activities in this direction, we shall feel very much rewarded. It is hoped that the form of presentation will be found useful in indicating the kind of information which has been made available by research workers up to the present day. Chemical engineering is in its infancy, and we must not suppose that the approach presented here must necessarily be looked upon as correct in the years to come. One of the advantages of this subject is that its boundaries are not sharply defined.

Finally, we should like to thank the following friends for valuable comments and suggestions: Mr. G. H. ANDERSON, Mr. R. W. CORBEN, Mr. W. J. DE COURSEY, Dr. M. GUTER, Dr. L. L. KATAN, Dr. R. LESSING, Dr. D. J. RASBASH, Dr. H. SAWISTOWSKI, Dr. W. SMITH, Mr. D. TRAIN, Mr. M. E. O'K. TROWBRIDGE, Mr. F. E. WARNER and Dr. W. N. ZAKI.

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# INTRODUCTION

THE understanding of the design and construction of chemical plant is frequently regarded as the essence of chemical engineering. Starting from the original conception of the process by the chemist, it is necessary to understand the chemical, physical and many of the engineering features in order to develop the laboratory process on an industrial scale. In this volume, we shall be mainly concerned with the physical nature of the processes that take place in industrial units, and, in particular, with determining the factors that influence the rate of transfer of material. The basic principles of these operations, namely fluid dynamics, and heat and mass transfer, have been discussed in Volume 1, and it is the application of these principles that forms the main part of this volume.

Throughout what are conveniently regarded as the process industries, there are many physical operations that are common to a number of the individual industries, and, as explained in Volume 1, these are regarded as unit operations. Thus, the separation of solids from a suspension by filtration, the separation of liquids by distillation, or the removal of water by evaporation and drying are typical operations of this kind. The problem of designing a distillation unit for the fermentation industry, the petroleum industry or the organic chemical industry is, in principle, the same, and it is mainly in the details of construction that the differences will occur. The concentration of solutions by evaporation is again a typical operation that is basically similar in the handling of sugar, or salt, or fruit juices, though there will be differences in the most suitable arrangement. This form of classification has been used here, but we have grouped the operations according to the mechanism of the transfer operation, so that the diffusional processes of distillation, absorption and liquid-liquid extraction are taken in successive chapters, and the operations involving solids in fluids are considered together. In examining many of these unit operations, we shall find that the rate of heat transfer or the nature of the fluid flow is the governing feature. The transportation of a solid or a fluid stream is another instance of the importance of understanding fluid dynamics.

One of the difficult problems of design is that of maintaining conditions of similarity between laboratory units and the larger industrial plants. Thus, if a mixture is to be maintained at a certain temperature during the course of an exothermic reaction, then on the laboratory scale there is rarely any real difficulty in maintaining isothermal conditions. On the other hand, in a large reactor the ratio of the external surface to the volume—which is inversely proportional to the linear dimension of the unit—is in most cases of a different order, and the problem of removing the heat of reaction becomes a major item in design. Some of the general problems associated with scaling up are considered in Chapter 26 on mixing, and particular features occur in many chapters. Again, the introduction and removal of the reactants may present difficult problems on the large scale, especially if they contain corrosive liquids or abrasive solids. The general tendency with industrial units is to provide a continuous process, frequently involving a series of stages. Thus, exothermic reactions may be carried out in a series of reactors with interstage cooling between them.

The planning of a process plant will involve the determining of the most economic method, and later the most economic arrangement of the unit operations used in the process. This amounts to designing a chemical process to provide the best combination of capital and operating costs. We have not in this volume considered the question of costs in any great detail, but we have aimed at indicating the conditions under which various types of units will operate in the most economical manner. Without a thorough knowledge of the physical principles involved in the various operations, it will not be possible to select the most suitable one for a given process. This aspect of the design can be considered by taking one or two simple illustrations of separation processes. The particles in a solid-solid system may be separated, first according to size, and secondly according to the material. Generally, sieving is the most satisfactory method of classifying relatively coarse materials according to size, but the method is impracticable for very fine particles and a form of settling process is generally used. In the first of these processes, we are directly utilising the size of the particle as the basis for the separation, and, in the second, we are dependent on the variation with size of the behaviour of particles in a fluid. A mixed material can also be separated into its components by means of settling methods, because the shape and density of particles also affect their behaviour in a fluid. Other methods of separation depend on differences in surface properties (froth flotation), magnetic properties (magnetic separation), and on differences in solubility in a solvent (leaching). For the separation of miscible liquids, the three commonly used methods are:

1. Distillation—depends on difference in volatility.

2. Liquid-liquid extraction—depends on difference in solubility in a liquid solvent.

3. Refrigeration—depends on differences in melting point.

This problem of selecting the most appropriate operation will be further complicated by such factors as the concentration of liquid which gives rise to crystals. Thus, in the separation of a mixture of ortho-, meta-, and para-mononitrotoluenes, the decision must be made as to whether it is better to carry out the separation by distillation followed by crystallisation, or in the reverse order. The same kind of consideration will arise when we are concentrating a solution of a solid; we must decide whether to stop the evaporation process when a certain concentration of solid has been reached and then to proceed with filtration followed by drying, or whether to continue the concentration by evaporation to such an extent that we can leave out the filtration stage and go straight on to drying.

In many operations, for instance in a distillation column, it is necessary to understand the fluid dynamics of the unit, as well as the heat and mass transfer relationships. These factors are frequently interdependent in a complex manner, and it is essential to consider the individual contributions of each of the mechanisms. Again, in a chemical reaction the final rate of the process may be governed by a heat transfer process or by the chemical kinetics, and it is essential to decide which is the controlling factor.

Some indication of the method of using the information on unit operations can be obtained by considering the example of a sulphuric acid plant. First, it is necessary to select the process and the raw materials which are to be used. It will be supposed that the process involves burning sulphur to sulphur dioxide, followed by oxidation to sulphur trioxide by what is known as the contact process, and that it is proposed to employ rock sulphur, oxygen from the atmosphere, and water. Alternative sources of sulphur include iron pyrites and spent oxide from gas works, but, although these are considerably cheaper than rock sulphur, it is necessary to install additional equipment for cleaning the gas. Secondly, we must study the kinetics of the chemical reactions. Here we are concerned basically with the following three reactions:

(i) $S + O_2 = SO_2 \quad (\Delta H = -70{,}920 \text{ lb-cal/lb-mol at } 25^\circ\text{C})$.

(ii) $2SO_2 + O_2 = 2SO_3 \quad (\Delta H = -47{,}500 \text{ lb-cal/lb-mol at } 420^\circ\text{C})$.

(iii) $SO_3 + H_2O = H_2SO_4 \quad (\Delta H = -32{,}000 \text{ lb-cal/lb-mol at } 100^\circ\text{C})$.

The overall reaction is therefore

$$2S + 3O_2 + 2H_2O \rightarrow 2H_2SO_4.$$

Each of the reactions is exothermic, and the successful and economic operation of the plant depends on the removal and subsequent utilisation of the heats of reaction. The first reaction is a simple gas-phase combustion which goes virtually to completion under adiabatic conditions, and it is not necessary therefore to remove heat in the combustion chamber itself. The second reaction does not proceed sufficiently rapidly except in the presence of a catalyst, generally vanadium pentoxide dispersed on a filler, and it becomes self-supporting at temperatures above about 400°C. Because the reaction is strongly exothermic, the equilibrium point is adversely affected by rise in temperature, and, because this effect is very marked, the reaction should be carried out as nearly as possible under isothermal conditions. The reaction rate is high in the presence of the catalyst, and the reaction can be regarded as proceeding to completion at each stage. The composition of the final gas can be calculated as a function of temperature, and therefore the composition of the equilibrium mixture at each adiabatic stage can be determined because all the heat of reaction appears in the products. The temperature of the outlet gases from each successive reactor will be lower, and therefore the $SO_3$ content will rise. The third reaction involves absorption of $SO_3$ in water and an immediate chemical reaction to produce $H_2SO_4$. As this reaction is exothermic, there would be a considerable rise in temperature and mist formation if it were carried out in this way. In practice, the heat capacity of the liquid phase is increased by absorbing the $SO_3$ in 98% $H_2SO_4$ rather than in water.

It can be seen from these considerations that a thorough understanding of the physical processes taking place in the individual units of chemical plant is an essential requirement in the training of a chemical engineer. It is only by understanding the mechanisms, and then the method of constructing the equipment, that it will be possible to obtain a complete picture, including the cost of the operation.

## Section E

# *Flow of Fluids past Particles*

## CHAPTER 11

# *Flow of Fluids through Granular Beds and Packed Columns*

In Volume 1, we evolved methods of determining the relation between pressure drop and velocity for the flow of fluid through regular pipes and ducts. In many cases, however, we are concerned with the flow through irregular channels, such as those formed between the particles in a bed of granular material.

In the filtration of a suspension, for example, liquid flows at a relatively low velocity through the spaces between the particles which have been retained by the filter medium and, as a result of the continuous deposition of solids, the resistance to flow increases progressively throughout the operation. In these conditions the channels are usually quite small and the velocity low, so that streamline flow is obtained.

On the other hand, packed columns containing relatively larger materials are used in order to promote turbulence and to increase the area of contact between two immiscible fluids. Here the pressure difference over the column is influenced by the rate of flow of each of the two phases. Packed columns are extensively used in gas absorption where one constituent is removed from a gas by means of a liquid flowing countercurrently. They are also used as fractionating columns in distillation plant and for solvent extraction.

Whereas the purpose of the filter press is very different from that of the packed column, it should be appreciated that they present similar hydraulic problems. In neither case is the problem capable of a complete analytical solution.

### FLOW OF FLUIDS THROUGH BEDS OF PARTICLES—STREAMLINE FLOW

The first experimental work on the subject was carried out by DARCY[7] in 1830 in Dijon when he examined the rate of flow of water from the local fountains through beds of sand of various thicknesses. He showed that the average velocity, as measured over the whole area of the bed, was directly proportional to the driving pressure and inversely proportional to the thickness of the bed. This relation, which has subsequently been confirmed by a number of workers, can therefore be written as follows—

$$u = K \, . \, \frac{\Delta P}{l} \qquad \ldots .(11.1)$$

(where $\Delta P$ is the pressure difference across the bed,

$l$ is the thickness of the bed,

$u$ is the average velocity of flow of the fluid, defined as $\frac{1}{A} \cdot \frac{dV}{dt}$,

$A$ is the total cross-sectional area of the bed,

$V$ is the volume of fluid flowing in time $t$,

$K$ is a constant depending on the physical properties of the bed and the fluid.)

The linear relation between the rate of flow and the pressure difference leads one to suppose that the flow was streamline. This would be expected because the Reynolds Number for the flow through the pore spaces in a granular material is low since both the velocity of the fluid and the width of the channels are normally small. Equation 11.1 can be compared with the expression for the mean velocity of a fluid flowing in a circular pipe; this has already been derived in Chapter 3 (equation 3.16) and may be written

$$u_1 = \frac{d_1^2}{32\mu} \frac{\Delta P}{l'} \qquad \ldots.(11.2)$$

where $\mu$ is the viscosity of the fluid,

$u_1$ is the mean velocity of the fluid,

$d_1$ is the diameter of the pipe and

$l'$ is the length of the pipe.

If the free space in the bed is assumed to consist of a series of tortuous channels, we can rewrite equation 11.2 for flow through a bed as

$$u_1 = \frac{d_m'^2}{K'\mu} \frac{\Delta P}{l'} \qquad \ldots.(11.3)$$

where $d_m'$ is some equivalent diameter of the pore spaces

and $K'$ is a dimensionless constant whose value depends on the structure of the bed.

This treatment is not strictly justified because the individual channels are really part of an interconnected series of passages.

An important property of the bed, which has a very large effect on its resistance to the flow of fluid, is the porosity or voidage, denoted by the symbol $e$. This is defined as the proportion of the total volume of the bed which is accounted for by the free space between the particles. The looser the packing, the lower will be the resistance to flow. If the particles are randomly packed, the voidage should be approximately constant throughout the bed and the resistance to flow the same in all directions.

It will be shown on page 392, in a bed of randomly distributed particles of voidage $e$, the average free cross-sectional area in any plane is $1/e$ of the total cross-sectional area.

Thus the average velocity, as measured over the whole cross-sectional area of the bed, will be $e$ times the average velocity in the capillary spaces,

i.e. $$u = u_1 e \qquad \ldots.(11.4)$$

Further, the length of the flow passages will be greater than the thickness of the bed but a direct proportionality would be expected between these two quantities,

i.e. $$l \propto l'$$

Substituting in equation 11.3,

$$\frac{u}{e} = \frac{d_m'^2}{K''\mu} \frac{\Delta P}{l} \qquad \ldots.(11.5)$$

The hydraulic mean diameter, which is defined as four times the ratio of the cross-sectional area to the wetted perimeter, has been found suitable for representing the equivalent diameter of a non-circular channel for turbulent flow conditions (see Vol. 1, page 47). Though the flow is streamline here, KOZENY⁽¹⁷⁾ has suggested that a function of the hydraulic mean diameter is used in place of $d_m'$ in equation 11.5 and that we write

$$d_m' = \frac{\text{volume of the voids}}{\text{total surface of the material forming the bed}}$$

$d_m'$ will be seen to be equal to one-quarter of the hydraulic mean diameter. For unit volume of bed, the pore space is $e$ and the exposed surface of the particles is $(1 - e)S$, where $S$ is the specific surface of the material (i.e. its surface per unit volume).

Thus $$d_m' = \frac{e}{(1-e)S} \qquad \ldots.(11.6)$$

Substituting in equation 11.5,

$$\frac{u}{e} = \frac{1}{K''\mu} \frac{e^2}{S^2(1-e)^2} \frac{\Delta P}{l}$$

i.e. $$u = \frac{1}{K''} \frac{e^3}{(1-e)^2} \frac{1}{\mu S^2} \frac{\Delta P}{l} \qquad \ldots.(11.7)$$

The commonly accepted value of $K''$, often known as KOZENY'S constant, is 5, but COULSON⁽⁴⁹⁾ has shown that it is a function of the porosity, the particle shape, the orientation of the particles and the size range. In Fig. 11.1 the variation of $K''$ with $e$ is shown for a number of different shapes of material. The value of 5 appears to be a fair average for beds of granular material and for isometric particles generally; for random spheres, there is no orientation effect, of course. With spheres, the range of voidage obtainable in practice is very small—from about 0·4 to 0·55—whereas the theoretical range with stable arrangements of spheres is from 0·28 to 0·78, the extreme values being obtained only as a result of careful arrangement of the individual particles. With most granular materials the voidage lies between about 0·3 and 0·5, but values of 0·9 are obtained with specially designed packings, such as rings. The large variation of $K''$ obtained by COULSON is not reported by other workers and may be attributable to the relatively large particles which he used. Recent work by MARTIN(59) has provided evidence that the resistance of beds of specially orientated spheres may depend on the direction of flow. The largest range of values of $K''$ is for cylinders and flat plates for which $K''$ varies from about 3 to 6, according to their orientation.

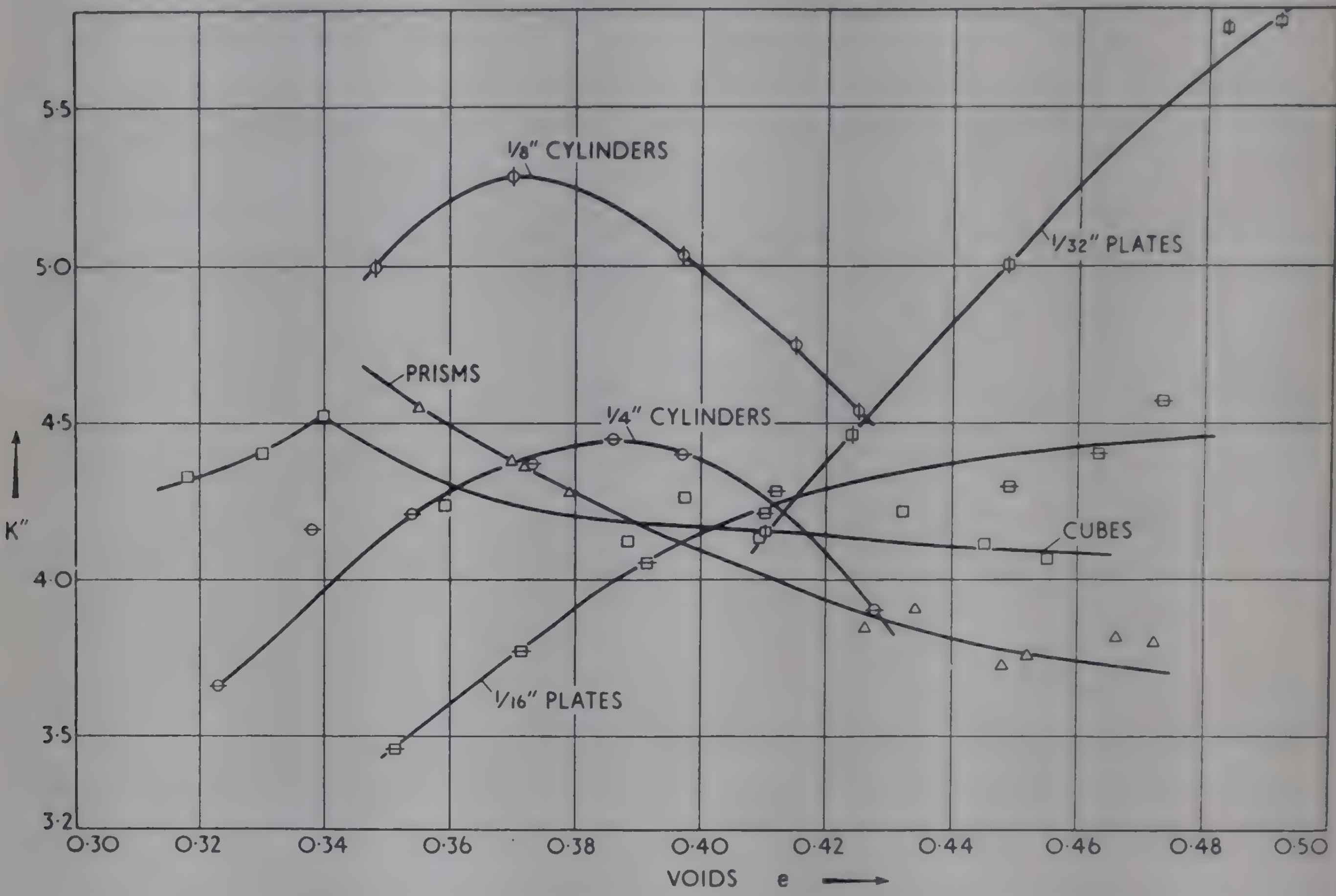


Fig. 11.1. Variation of KOZENY's constant $K''$ with voidage for various shapes

The resistance to flow is a function of the shape and size of the free spaces and therefore is not definable in terms of the particle size and voidage alone. The resistance will be a function of the average angle subtended by the surfaces of the particles with the mean direction of flow of the fluid, and the introduction of an appropriate factor has met with a limited success in the correlation of data. Accurate measurements of the orientation of particles in a randomly packed bed were made by KIHN(35) who poured molten wax over the particles so that they maintained their relative positions and then examined the orientation, layer by layer.

Again, the above treatment does not take account of the fact that some of the contacts between particles will be area contacts and part of the surface of the particles will thus be rendered ineffective in offering resistance to flow.

Most of the experimental work on the flow of fluids through beds has been concerned with beds of particles of uniform size. The addition of smaller particles to such a bed can have two effects. First the voidage may be increased by the small particles getting between the larger particles and forcing them apart. Secondly, the smaller particles may decrease the voidage by filling the spaces between the larger ones. It is found experimentally that the tightest possible packing with particles of two different sizes is obtained when the ratio of the sizes is greater than 3. In general, the wider the range of particle sizes, the lower is the voidage obtainable.

The structure of the bed is markedly influenced by the nature of the surface on which the particles are deposited. Thus the initial conditions in a filtration operation may affect the structure of the whole filter cake. Fig. 11.2 shows the difference in orientation in a bed of cubical particles when formed on a plane surface (*a*) or on a layer of spheres (*b*).

The influence of the walls of the vessel containing the bed is important since the voidage is greater near the walls. ROSE(34, 37) considers the bed in two parts, a central core of voidage $e$, and an outer annulus of a higher voidage $e'$, where the close interlocking of the particles is prevented by the walls; the depth of the annulus is taken as one-half of the mean diameter of the particles. COULSON(49) has shown that for streamline flow the wall effect can be taken into account by the use of an empirical factor. For a given pressure difference, the velocity through an infinite bed is less than that in a finite bed by a factor $\left(1 + \frac{1}{2}\frac{S_c}{S}\right)^2$, where $S$ is the specific surface of the particles and $S_c$ is the surface of the containing vessel per unit volume of solid particles.

Although the resistance to flow cannot be expressed solely in terms of the particle sizes and the voidage, the expression obtained gives a clear indication that a small change in the voidage may make a large difference to the resistance. The rate of flow is proportional to $\frac{e^3}{(1-e)^2}$ (equation 11.7), and a change in the voidage from 0·5 to 0·6 will reduce the resistance by a factor of 2·7. A number of workers have expressed the function of $e$ as a simple power function but this does not give such a satisfactory correlation of the experimental data.

The resistance of the bed may change as the result of the passage of fluid through it. Thus, if the arrangement of the particles is not very stable, channelling may occur and the fluid will pass preferentially at certain sections where the resistance to flow is less than the average value. The overall resistance of the bed will thus fall and the channels will tend to open out with the continued passage of the fluid because of its eroding effect. In many cases the flow through beds of fine particles is uneven because of the action of surface tension forces in the smallest of the pores.

Equation 11.7 is the most satisfactory equation for calculation of the rate of flow of fluid under streamline conditions through a bed, though it is seen that some error may be introduced if the commonly accepted value of 5 is used for the constant $K''$. The main deviations from this value appear to occur with beds composed of flat plates or cylinders, as already indicated, but in other cases $K''$ is almost constant; this applies also to flow through beds of broken granular material. Equation 11.7 is sufficiently reliable for the calculation of the specific surface of a powder from measurements of the flow rates for various pressure drops across a bed of the material. In this case a measure will be obtained of the effective surface of the particles, re-entrant surfaces not contributing in the same way to the resistance to the flow. No account has been taken of the expansion of the fluid as it passes through the bed and therefore equation 11.7 refers only to small pressure changes for compressible fluids.

## Average Free Cross-Sectional Area

It has so far been assumed that the average free cross-sectional area in a bed of voidage $e$ is also equal to $e$. This assumption will now be justified.

Consider a cube of granular bed of side $l$, containing $N$ particles of characteristic linear dimension $D$. Then the total volume of the bed is $l^3$ and the total volume of particles is $Nk'D^3$, where $k'$ is a constant, whose value depends on the shape of the particle.

The voidage of the bed $e$ is therefore

$$1 - \frac{Nk'D^3}{l^3} \qquad \ldots.(11.8)$$

Now assume that the particles are randomly distributed in the bed and that the cube is cut by a plane parallel to one of its faces. Then the statistical probability of cutting any particular particle is approximately $\frac{D}{l}$ and the average number of particles cut by the plane is $\frac{ND}{l}$.

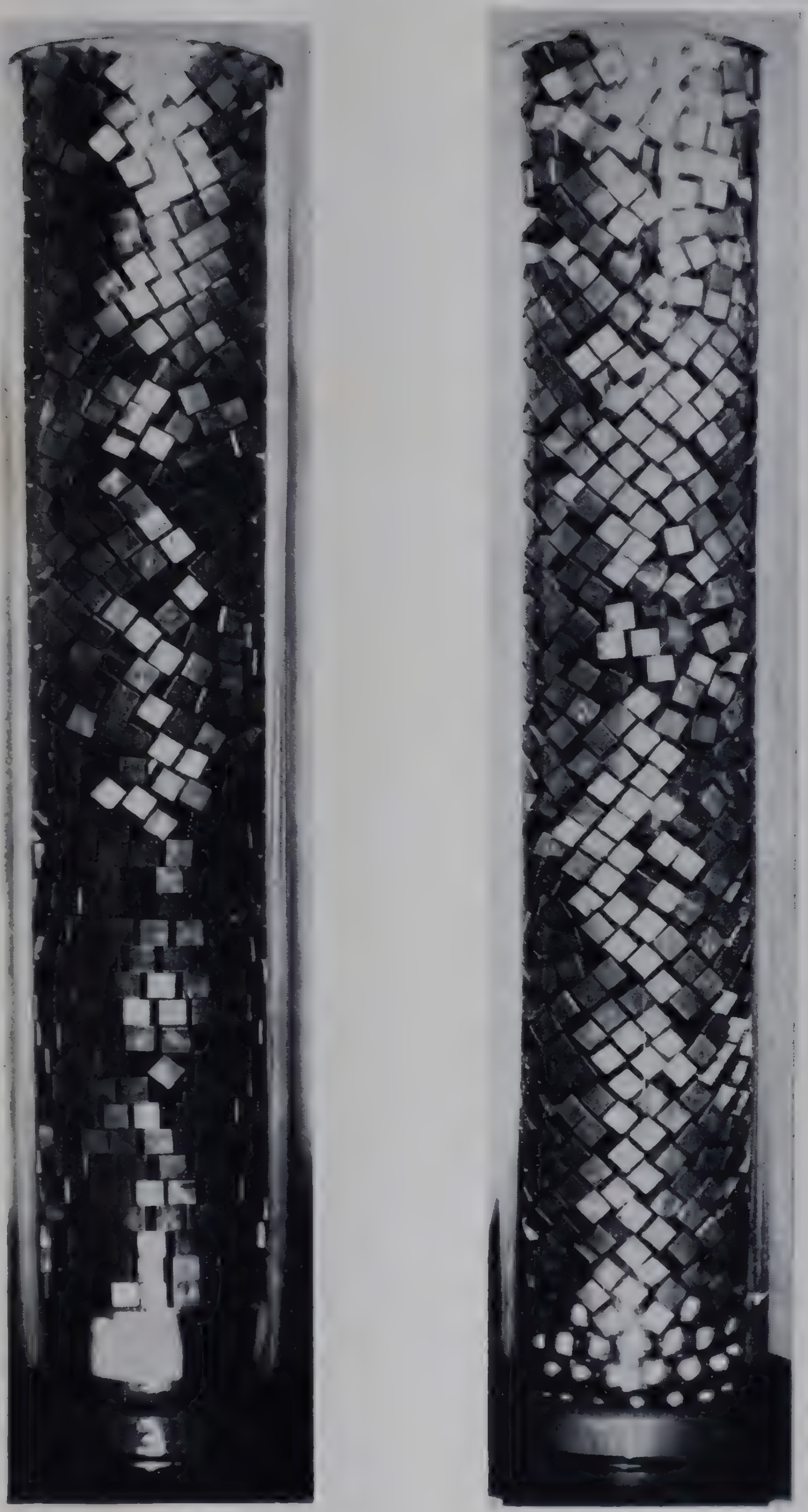
The average area of each particle exposed as a result of cutting with the plane is approximately equal to the volume of the particle divided by its characteristic linear dimension,

i.e. $$\frac{k'D^3}{D} = k'D^2$$

Thus the average area of particles exposed as a result of cutting by the plane

$$= k'D^2 \, . \, \frac{ND}{l}$$

$$= Nk'\frac{D^3}{l}$$



(a) (b)

Fig. 11.2. Packing of cubes, stacked on (a) plane surface, and (b) on bed of spheres

Since the total cross-sectional area is $l^2$, the average free cross-sectional area is

$$l^2 - Nk' \frac{D^3}{l}$$

The fraction of the cross-sectional area available for flow is therefore

$$\frac{l^2 - Nk' \dfrac{D^3}{l}}{l^2} = 1 - Nk' \frac{D^3}{l^3}$$

$$= e \quad \text{(from equation 11.8)}$$

## STREAMLINE AND TURBULENT FLOW THROUGH GRANULAR BEDS

The first attempt at using dimensionless groups to correlate data on the flow of fluid through beds composed of granular particles was made by **Blake**(11), who expressed the size of his particles in terms of their specific surface $S$. Modifications of his method are now commonly in use and these involve plotting a dimensionless resistance factor against a modified Reynolds Number. The most satisfactory method is that adopted by **Carman**(29) and others, whe the modified Reynolds Group $Re.'$ incorporates the density and viscosity c he fluid, the mean velocity in the voids themselves $u_1$, and a characteristic li dimension of the channels $d'_m$, which is taken as the ratio of the volume c e channels to the total surface of the particles, as in **Kozeny**'s treatment.

Now $$u_1 = \frac{u}{e} \qquad \text{(from equation 11.4)}$$

and $$d'_m = \frac{e}{(1 - e) . S}, \qquad \ldots .(11.6)$$

so that $$Re.' = \frac{\dfrac{u}{e} \dfrac{e}{(1 - e)S} \rho}{\mu}$$

$$= \frac{u . \rho}{S . \mu(1 - e)} \qquad \ldots .(11.9)$$

The friction factor which is plotted against the modified Reynolds Number is $R'/\rho u_1^2$, where $R'$ is the component of the drag force per unit area of particle surface, in the direction of motion. Now consider the forces acting on the fluid in a bed of unit cross-sectional area and thickness $l$. The volume of particles in the bed is $l . (1 - e)$ and therefore the total surface is $S . l . (1 - e)$. Thus the resistance force $= R' . S . l . (1 - e)$. This force on the fluid must be equal to that produced by a pressure drop of $\Delta P$ across the bed. Then, since the free cross-section of fluid is equal to $e$,

$$\Delta P \,.\, e = R' \,.\, l \,.\, S \,.\, (1 - e)$$

and

$$R' = \frac{\Delta P \,.\, e}{l \,.\, S \,.\, (1 - e)} \qquad \ldots.(11.10)$$

Thus

$$\frac{R'}{\rho u_1^2} = \frac{\Delta P \,.\, e^3}{l \,.\, S \,.\, (1 - e)\rho u^2} \qquad \ldots.(11.11)$$

When $R'/\rho u_1^2$ is plotted against $Re.'$ using logarithmic co-ordinates, it is found that the data for the flow through randomly packed beds can be correlated by a single curve (Fig. 11.3). This curve can be divided into three portions. At low values of $Re.'$, it is a straight line of slope equal to $-1$. For values of $Re.'$ between about 2 and 100 the slope gradually changes from $-1$ to $-\frac{1}{4}$, and at values of $Re.'$ in excess of about 100 it is approximately a straight line of slope $-\frac{1}{4}$.

Under streamline conditions, we have,

$$u = \frac{e^3}{K''(1 - e)^2} \cdot \frac{1}{\mu S^2} \cdot \frac{\Delta P}{l} \qquad \ldots.(11.7)$$

so that

$$\frac{\Delta P}{l} = \frac{(1 - e)^2}{e^3} K'' S^2 \mu u.$$

Thus

$$\frac{R'}{\rho u_1^2} = \frac{(1 - e)^2}{e^3} \,.\, K'' S^2 \mu u \,.\, \frac{e^3}{S(1 - e)\rho u^2}$$

$$= \frac{K''(1 - e) \,.\, \mu \,.\, S}{u \,.\, \rho}$$

$$= K'' Re.'^{-1} \qquad \ldots.(11.12)$$

As already indicated, $K''$ has an average value of about 5. This equation is applicable for values of $Re.'$ less than about 2 when the flow is streamline. At higher values, the flow starts to become turbulent in the largest channels and subsequently turbulence sets in in successively smaller channels as the value of $Re.'$ increases. Hence there is only a very gradual change in the slope of the curve from $-1$ to $-\frac{1}{4}$. It is probable that the flow never becomes completely turbulent, since some of the passages may be so small that streamline conditions prevail even at very high flow rates.

FORCHHEIMER⁽²⁾ has suggested that the resistance to flow should be considered in two parts; that due to the viscous drag at the surface of the particles, and that due to loss in turbulent eddies and at the sudden changes in the cross-section of the channels,

i.e.

$$\Delta P = \alpha u + \alpha' u^n \qquad \ldots.(11.13)$$

The first term in this equation will predominate at low rates of flow where the losses are mainly attributable to skin friction, and the second term will become significant at high flow rates and in very thin beds where the enlargement and contraction losses become very important. At very high flow rates the effects of viscous forces are negligible.

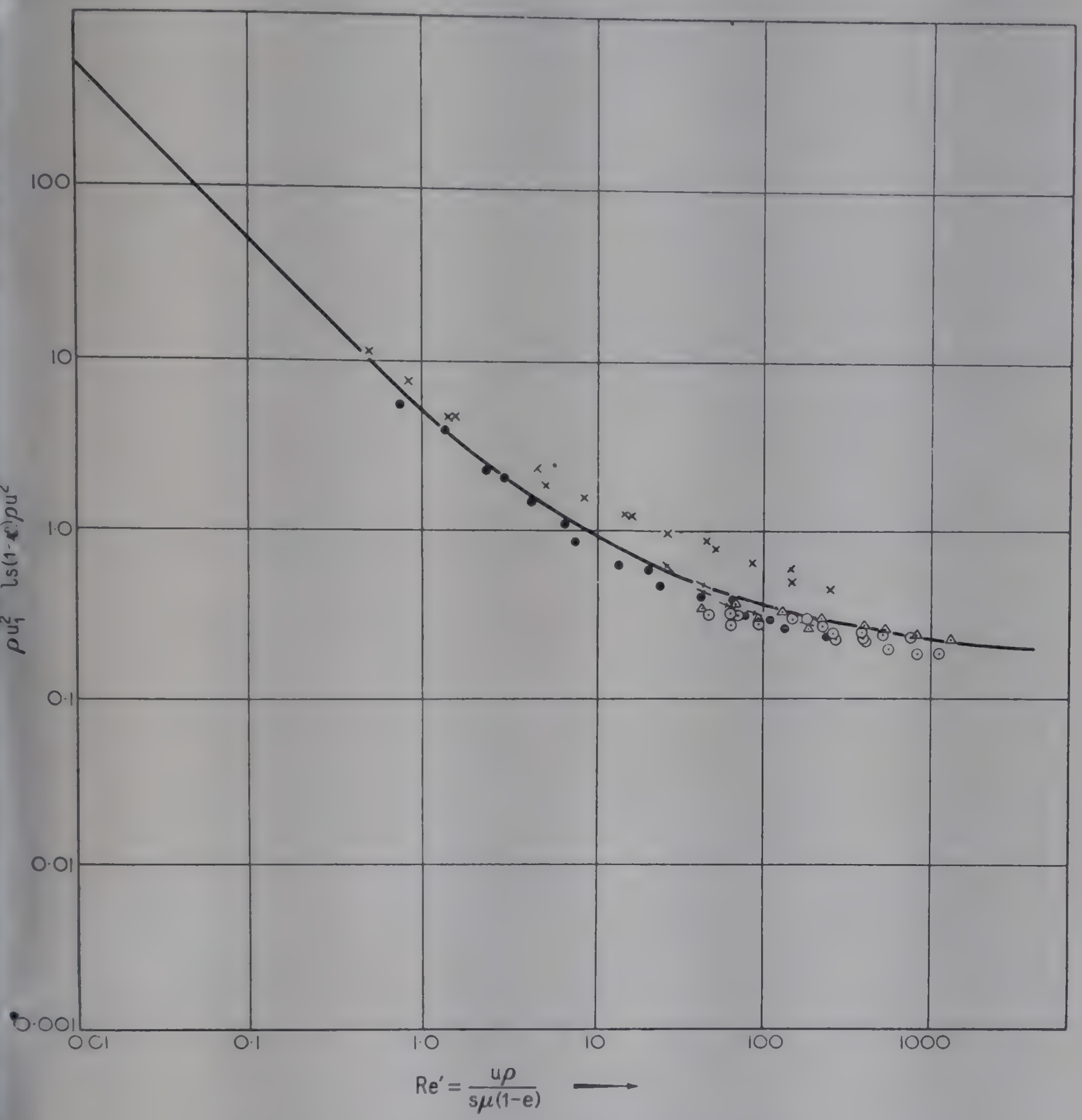


Fig. 11.3. Carman's graph of $\frac{R'}{\rho u_1^2}$ against *Re.'*

Carman[(29)] has shown that the available data on pressure drops for various rates of flow through beds are correlated by the following equation over the whole range of values of the Reynolds Group investigated.

$$\frac{R'}{\rho u_1^2} = 5\,Re.'^{-1} + 0{\cdot}4\,Re.'^{-0{\cdot}1} \qquad \ldots.(11.14)$$

At very high rates of flow it is found that the roughness of the particles has a small effect on the frictional force, but this effect is appreciable only at rates exceeding those normally experienced in practice.

Chilton and Colburn(24), Rose(34, 37, 52) and others used a slightly different method of correlation and based their modified Reynolds Group on the mean velocity of flow and the diameter of the particles. This method of approach seems less logical because the fluid flows through the pore spaces and not through the particles, and it therefore appears more reasonable to use a characteristic dimension of the void space in the Reynolds Group. Further the velocity in the pore seems more relevant than the average velocity.

Chilton and Colburn were the first to use this method and they plotted the group $\frac{\Delta P}{l} \cdot \frac{d_p}{2\rho u^2}$ $(= \phi')$ against the Reynolds Group $\frac{u d_p \rho}{\mu}$ $(= Re.'')$. Here $d_p$ is the diameter of a spherical particle or the average diameter of a mixture of sizes or of non-spherical particles; the method of calculating the mean size is not stated. They suggested that for low rates of flow,

$$\phi' = 850\, Re.''^{-1} \qquad \text{....(11.15)}$$

and for high rates of flow,

$$\phi' = 38\, Re.''^{-0\cdot15} \qquad \text{....(11.16)}$$

The change from one law to the other is gradual, as would be expected, and occurs when $Re.''$ is between 40 and 80.

Rose used a similar correlation but plotted $\frac{\Delta P}{l} \cdot \frac{d_p}{\rho u^2}$ $(= \phi = 2\phi')$ against the Reynolds Group $\frac{u d_p \rho}{\mu}$ $(= Re.'')$, and used an exponential function of the voidage, to take account of the shape of the free spaces. He specified that $d_p$ was the diameter of the sphere with the same specific surface as the material forming the bed. The results are correlated approximately by the equation

$$\phi = 1000\, Re.''^{-1} + 125 Re.''^{-\frac{1}{2}} + 14 \qquad \text{....(11.17)}$$

His curve for granular beds is shown in Fig. 11.4, in which the voidage function is arbitrarily taken as unity for a voidage of 0·40. It will be noted that Rose's coefficient $\phi$ is equal to $\frac{R'}{\rho u_1^{\,2}}$ multiplied by the factor $\frac{d_p S(1-e)}{e^3}$. The two methods of correlation generally correspond closely when the voidage is between about 0·35 and 0·4.

Morcom(38) has carried out work where the flow is intermediate between streamline and turbulent. He uses the same method of correlation as Rose, except that he takes his equivalent size as the diameter of the sphere with the same volume as the average particle. He gives the equations

$$\phi = 800\, Re.''^{-1} + 14 \qquad \text{....(11.18)}$$

for spheres and irregular broken materials

and

$$\phi = 1750\, Re.''^{-1} + 28 \qquad \text{....(11.19)}$$

for flat ended cylinders.

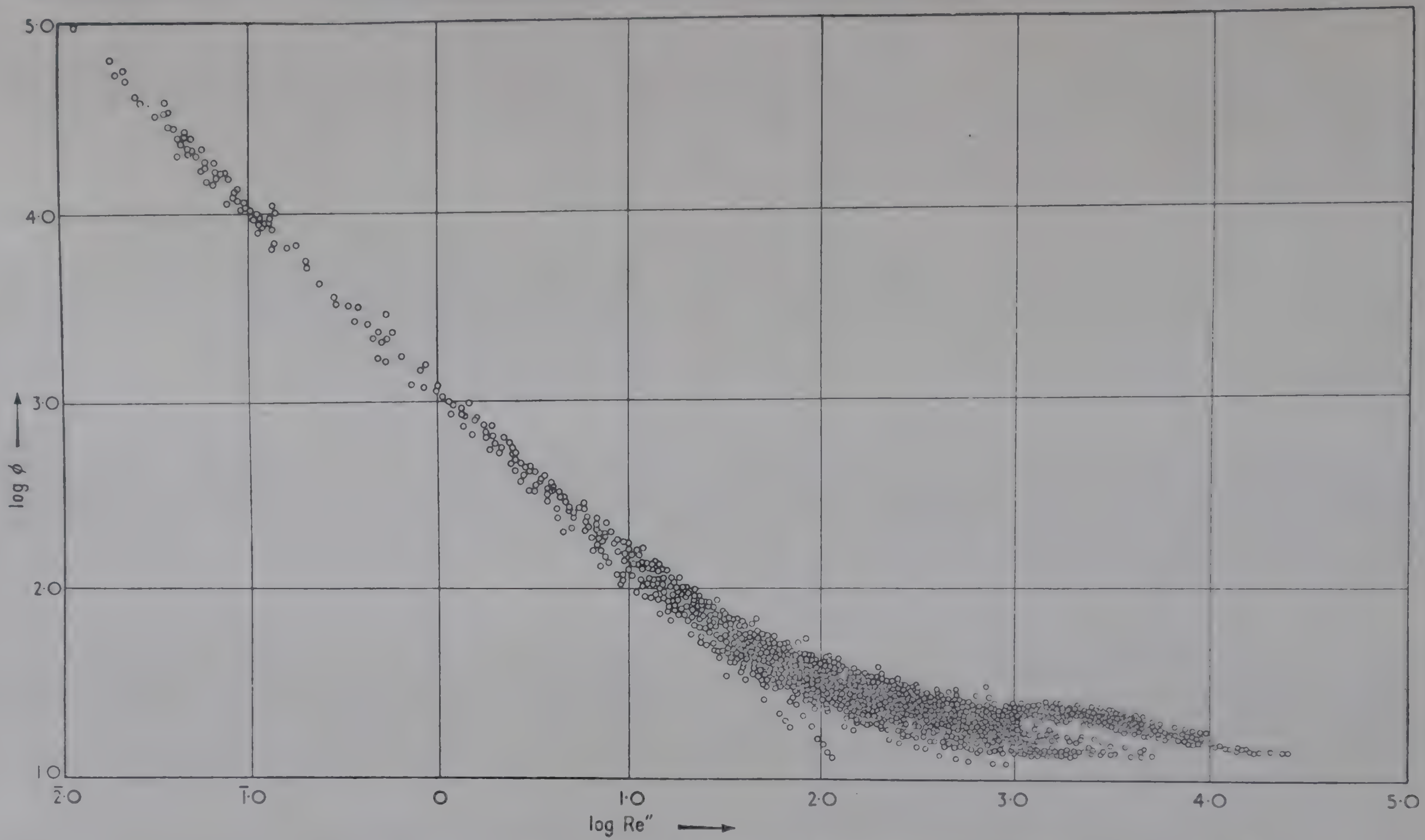


Fig. 11.4. ROSE's graph of log $\phi$ against log *Re.″* for granular material

It is of interest to note that both MORCOM and COULSON found anomalous results with cylinders. Equation 11.18 is in close agreement with equation 11.17 of ROSE.

More recently, BROWNELL and others$^{(44)}$ have attempted to use the ordinary friction chart (Fig. 3.3) for the flow of fluids in smooth round pipes to represent the data for the flow through beds of granular materials. The main difficulty is in the choice of suitable parameters to account for the nature of the flow spaces in terms of the voidage of the bed and the shape of the particles. The correlation is based on the use of the Reynolds Group $Re.'' = \frac{u d_p \rho}{\mu}$ and a modified friction factor $\frac{R''}{\rho u^2}$, defined by the equation

$$\Delta P = 4\left(\frac{R''}{\rho u^2}\right) \cdot \frac{l}{d_p}\ \rho u^2$$

which is identical in form with equation 3.7 for the flow through a pipe. In the above expressions, $u$ is the velocity of flow calculated over the total cross-section of the bed, and $d_p$ is the surface mean diameter of the particles. $Re.''$ is multiplied by a suitable parameter $f_1$ and $\frac{R''}{\rho u^2}$ divided by a second parameter $f_2$, both of which are so chosen that the plot of $Re.'' . f_1$ and $\frac{R''}{\rho u^2} \cdot \frac{1}{f_2}$ corresponds with the friction chart for a smooth pipe. The main difficulty in the method lies in assessing the correct values of the factors $f_1$ and $f_2$. BROWNELL has given curves of $f_1$ and $f_2$ as functions of voidage and "sphericity" $\psi$ and they are reproduced in Figs. 11.5 and 11.6. $\psi$ is defined by the relation

$$\psi = \frac{\text{surface area of the sphere having the same volume as the particle}}{\text{surface area of the particle}}.$$

These curves have been drawn from data obtained in BROWNELL'S own experiments and from data of other workers but the chief objection to the method is the inherent lack of reliability of the values of the parameters $f_1$ and $f_2$. In general, when the particles are randomly packed, high values of voidage are obtained only when the particles have a very low "sphericity."

BROWNELL has plotted $\frac{1}{f_2} . (R''/\rho u^2)$ against $f_1 . Re.''$ (Fig. 11.7) for a number of different materials and has claimed that the friction curve for a smooth pipe will correlate the data to an accuracy of about 6% over most of the range. In the region of the transition from streamline to turbulent flow conditions, however, deviations of up to 24% are found. This is explained by the fact that the transition from streamline to turbulent flow is quite sharp for pipe flow but takes place very gradually, as previously mentioned, for flow through granular beds. The deviations become serious at values of $Re.'' . f_1$ of about 400.

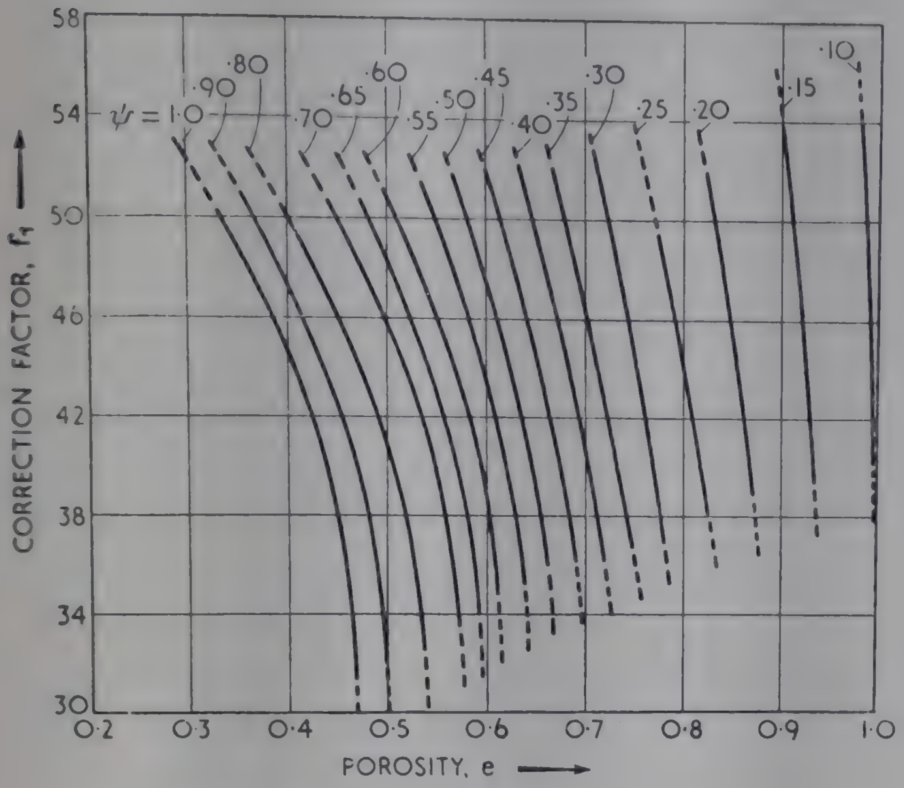


Fig. 11.5. Reynolds Number correction factor $f_1$

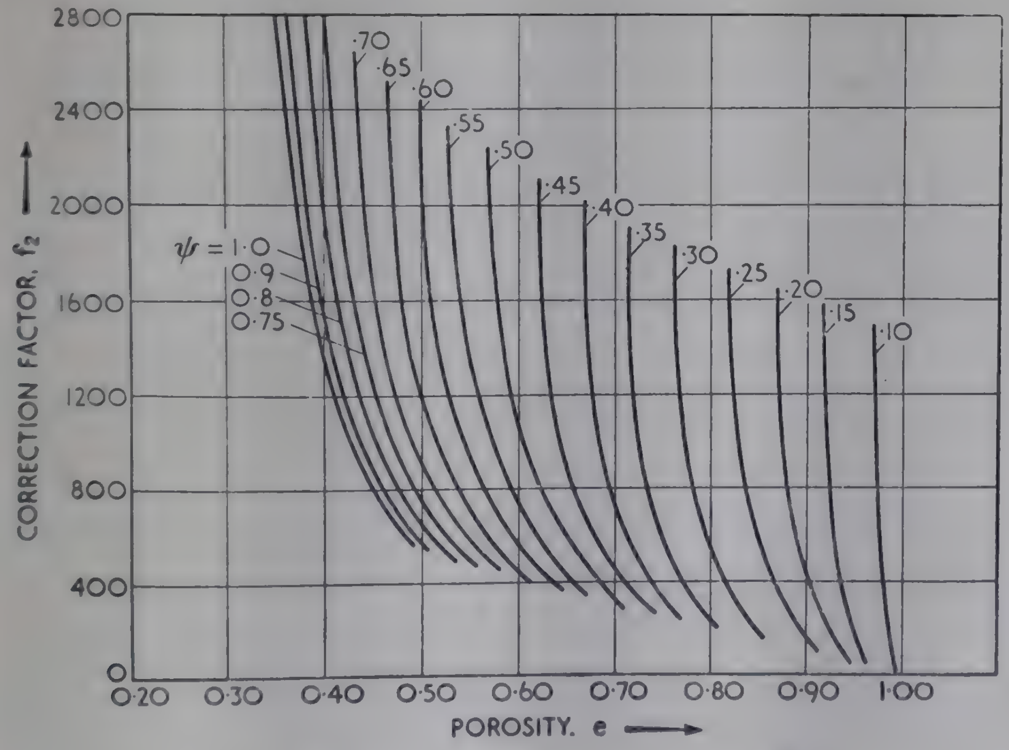


Fig. 11.6. Correction factor $f_2$ for $\frac{R''}{\rho u^2}$

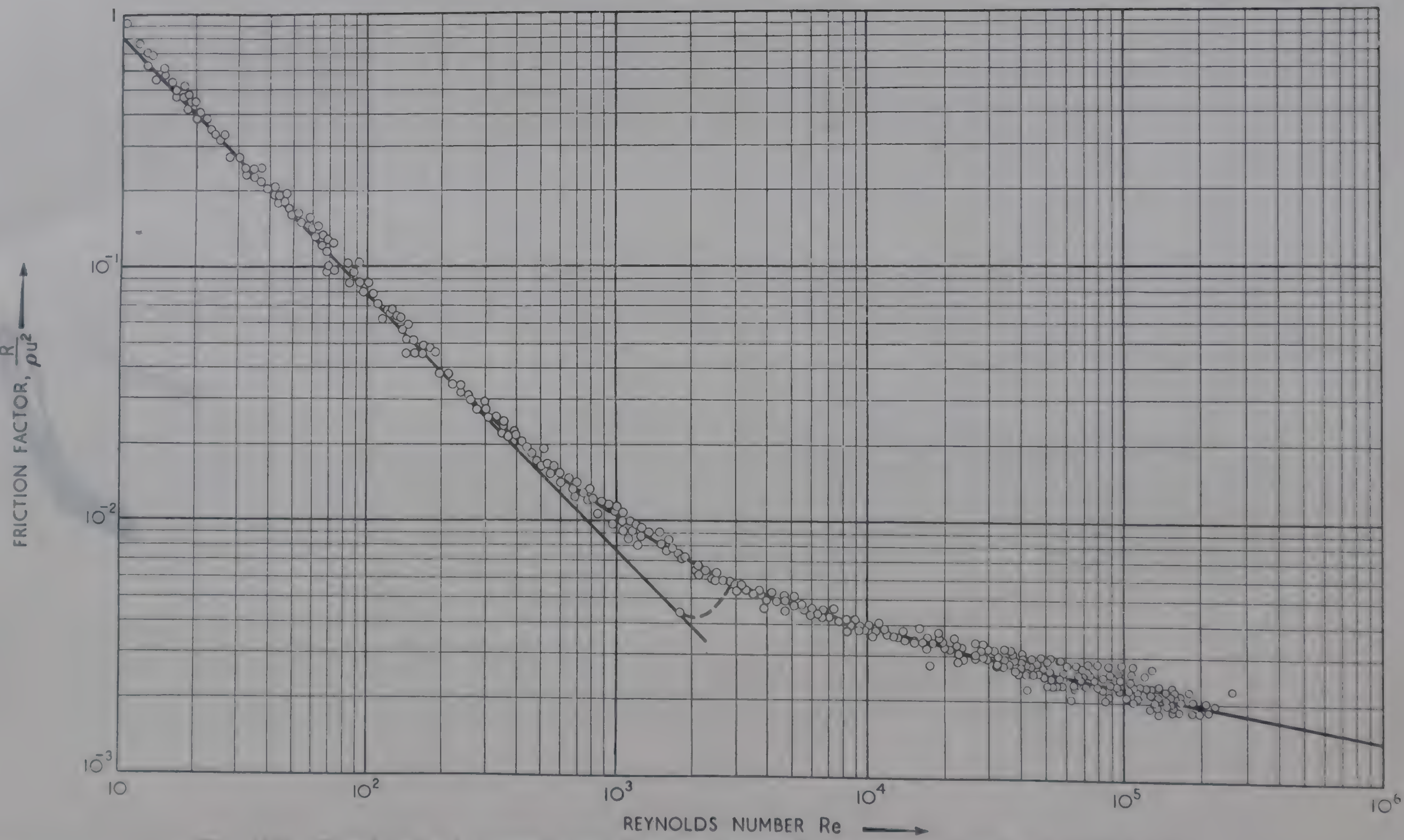


Fig. 11.7. Correlation of pressure drop for granular beds with friction chart for smooth pipe

## SIMULTANEOUS FLOW OF TWO FLUIDS(44)

The problem so far considered has been that of the flow of a single fluid through a bed of granular material and the whole of the void space is then available for flow by the one fluid. When two immiscible fluids are flowing simultaneously, the free space will be occupied partly by one fluid and partly by the other, and therefore the area available for the flow of either fluid will be reduced by the presence of the other. In general, one of the fluids will wet the packing and the other will be separated from it by a film of the first fluid. The fluids may be flowing co-currently or countercurrently and may consist either of two liquids or of a liquid and a gas. The problem of the countercurrent flow of a gas and a liquid, as in an absorption tower, will be discussed.

In the liquid-gas system, the liquid wets the solids and the gas passes through the void space which is not obstructed. Because there is a film of liquid on the particles, the shape of the passages will be modified considerably by the presence of the liquid and, in general, the surfaces of the channels will be smoother and the gas will not suffer such sharp changes in its direction of motion as with a dry packing material. Part of the volume of the bed will thus consist of solid particles, part of liquid, and part of gas.

The proportion of the void space which is occupied by liquid is known as the saturation $s$ of the bed. The quantity which defines the rate of flow of each of the fluids is the relative permeability. This is the ratio of the rate of flow of the fluid in question, to the corresponding rate of flow if that fluid filled the whole of the void spaces. WYCKOFF and BOTSET(28) measured the rates of flow of gas and oil through a bed composed of sand particles in the size range 70 to 120 mesh. The liquid was flowing in the downward direction and the gas upwards through the packing and liquid. The flow rates of gas and liquid were measured for a given pressure difference across the bed and various values of the liquid saturation $s$. The curves obtained for the relation between the relative permeability and the saturation $s$ are shown in Fig. 11.8. When the liquid saturation is less than about 0·18, the liquid is all held up in the packing and the gas flow is not affected by the presence of the liquid. At the other extreme, the gas flow ceases when the saturation is about 0·9. It therefore seems that certain of the voids are contributing, only to a very small extent, to the area available for the flow of either fluid and that the flow rates are not appreciably affected if these voids are obstructed. It is therefore suggested that some of the voids contain stationary liquid or gas and, for a given pressure drop across the bed, are not available for flow. The stationary liquid or gas in these voids is held there by surface tension forces. If a small void is filled with liquid which is bounded by gas at the inlet and the outlet to the void, a certain definite pressure drop, determined by the size of the void and the surface tension of the fluid, will be required before the liquid can be forced out. If the actual pressure drop is less than this, the liquid will be held in the void by surface tension forces, and that part of the free space of the bed will not be available for the flow of either fluid. Since the pressure difference required to force the liquid out of the void will be greater for the smaller voids, it is these voids which will not be available for flow. As the pressure drop across the bed is increased, the liquid will be forced from successively smaller voids and the proportion of the total voids containing stationary fluid will be reduced.

If a bed of granular material is initially saturated with liquid and a stream of gas is passed in an upward direction, it will gradually displace some of the liquid from the voids. When steady conditions have been reached, the gas will flow through the larger voids and the liquid will continue to flow as a film over the surface of the particles and through some of the smaller voids. If the pressure difference across the column is further increased, and the gas flow becomes larger, the liquid will be removed from more and more of the voids and, eventually, the liquid flow will cease. The gas flow under these conditions will be only slightly less than that through a dry bed for the same pressure difference. At this stage, the only liquid present will be that held in the smaller voids by surface tension forces.

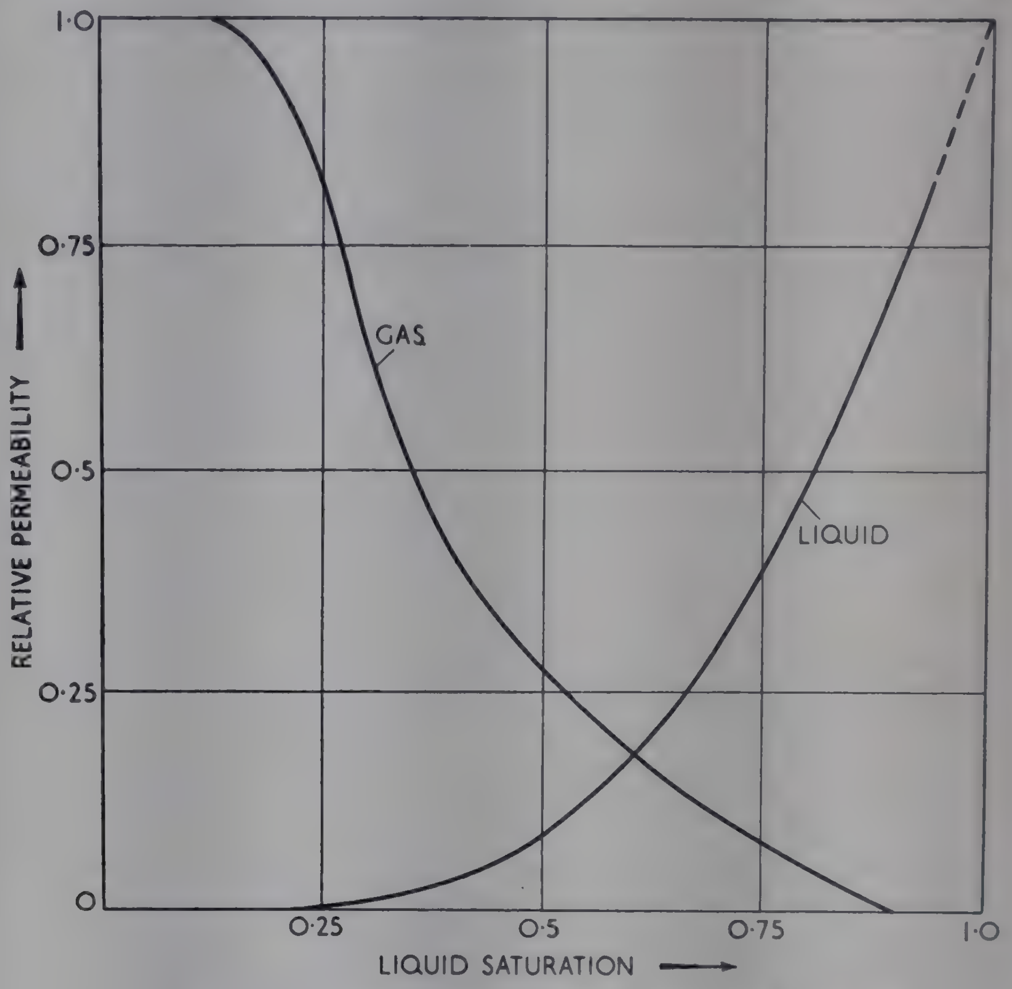


Fig. 11.8. Permeability of bed of sand to oil and gas

It is thus seen that the total volume of the bed can be divided into five parts as follows.

(1) The volume occupied by solid particles.
(2) That occupied by liquid in flow.
(3) That occupied by gas in flow.
(4) That containing stationary liquid.

(5) That containing stationary gas. This is usually a small proportion of the total volume and therefore can be neglected.

The distribution of the void space can conveniently be represented diagrammatically. The relative permeability of the bed to the liquid is plotted against the fraction of the total void space which is utilised in the various ways, for a given pressure drop over the bed in Fig. 11.9.

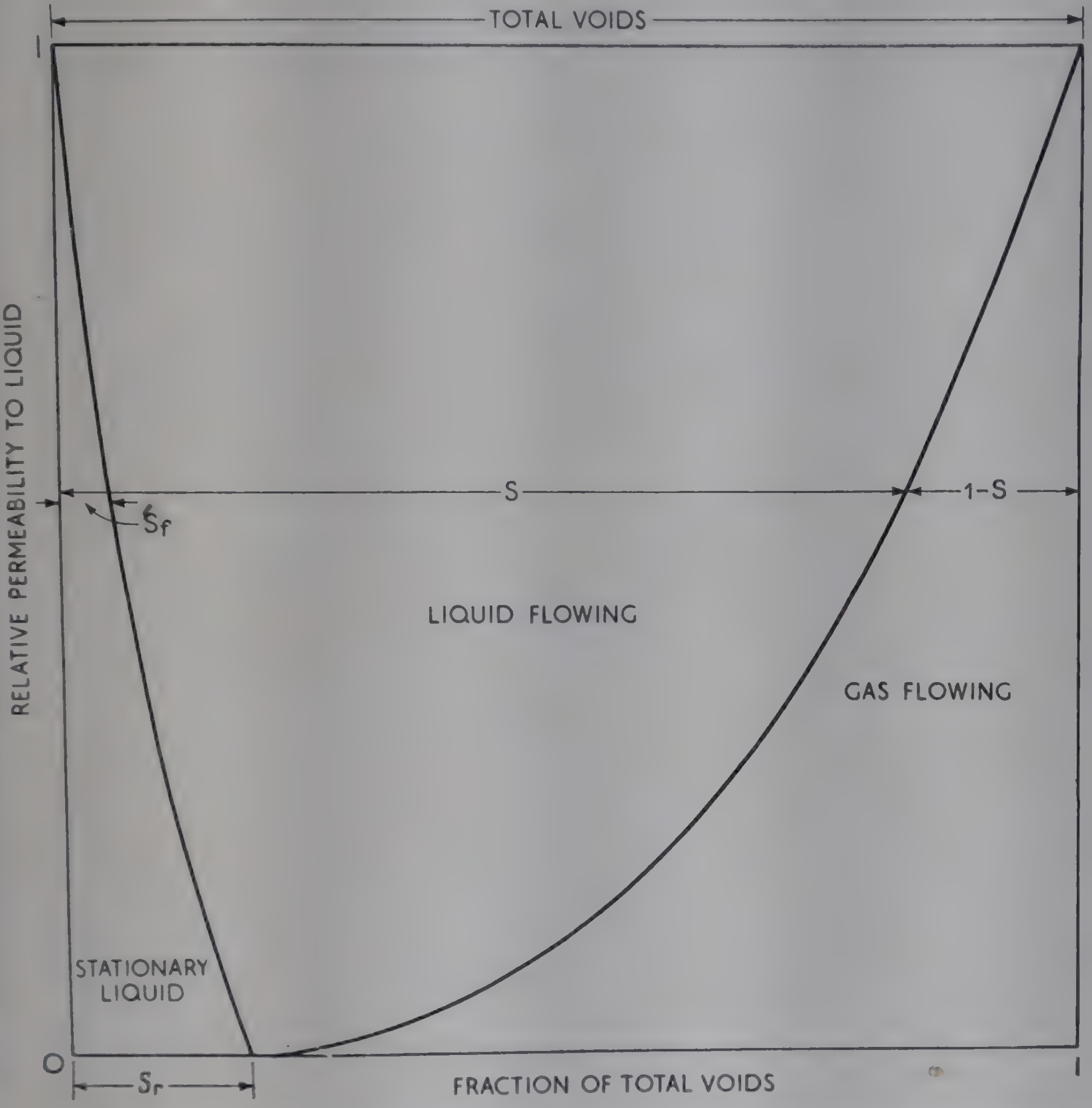

TOTAL VOIDS
1
RELATIVE PERMEABILITY TO LIQUID
S
1-S
Sf
LIQUID FLOWING
GAS FLOWING
STATIONARY LIQUID
O
Sr
FRACTION OF TOTAL VOIDS
1

Fig. 11.9. Distribution of fluids as function of relative permeability to liquid

The saturation $s$ represents the proportion of the voids which are occupied by liquid, either flowing or stationary. The residual saturation $s_r$ is the maximum proportion of the voids which can be eliminated from flow for a particular difference in pressure across the bed. The proportion of the voids in which fluid is stationary $s_f$ is assumed, as a first approximation, to be proportional to the volume of gas present in the bed, so that

$$s_f = s_r \frac{1 - s}{1 - s_r} \qquad \ldots.(11.21)$$

A further quantity, the effective saturation $s_e$, is the ratio of the voids in which flow of liquid is occurring to the total volume of voids in which flow of either fluid is taking place; that is to say, it is the saturation based on the useful volume of the voids. Thus

$$s_e = \frac{s - s_f}{1 - s_f}$$

$$= \frac{s - \left(\dfrac{1 - s}{1 - s_r}\right) . s_r}{1 - \left(\dfrac{1 - s}{1 - s_r}\right) . s_r}$$

$$= \frac{s - s_r}{1 - 2s_r + s . s_r} \qquad \text{....(11.22)}$$

The values of $s_f$ and $s_e$ can then be calculated for any value of $s$ provided the residual saturation $s_r$ is known. Now the residual saturation will be a function of the physical properties of the liquid (particularly of its surface tension), the pressure difference, which determines the rate of flow of gas, and the properties of the packing or particles. It is found that the residual saturation, determined experimentally over a range of conditions for thick beds of material —greater than 1 foot—, can be related to the dimensionless group $\mu_G u'/\sigma$, which is sometimes known as the capillary number *Ca*. In this group, $u'$ is the mean velocity of flow of the gas produced by the same pressure difference in a dry bed (calculated over the whole cross-section), $\sigma$ is the interfacial tension and $\mu_G$ is the viscosity of the gas. **Brownell**(44) has suggested that $s_r$ is given by the relation,

$$s_r = k'' . Ca.^{-0.26} \qquad \text{....(11.23)}$$

For thick beds, greater than about 1 ft, $k'' = 86$, approximately. For thin beds, up to about 2 in., such as encountered in filtration, $k'' = 40$ approximately.

In a packed bed, most of the fluid flows through the larger voids and the smaller ones make little contribution to the flow of either fluid so that the effective area available for flow of liquid will be approximately $s_e$ times that in a bed which is fully saturated. The value of the saturation $s$ corresponding to a given rate of flow of liquid can then be calculated, approximately, since

$$s_e = \frac{s - s_f}{1 - s_f} = \frac{s - s_r}{1 - 2s_r + s . s_r} \qquad \text{....(11.22)}$$

and $s_r$ can be estimated by the method given above.

The total volume of voids per unit volume of bed which will be available for the flow of the gas will be $(1 - s) . e$. Since the small voids will, under these conditions, be filled with liquid, it is the larger voids which are available for passage of gas. Now, in an unwetted bed, the whole of the free space will be available for gas flow but most of the flow will take place through the larger pores, so that, as a first approximation, flow can be considered to take place through a fraction $1 - s_r$ of the free space. Thus the effective void space for gas flow will be reduced by the presence of the liquid by a factor of about $\frac{1 - s}{1 - s_r}$. The problem is complicated, however, by the fact that the shape of the voids is changed by the wetting fluid so that the resistance per unit volume of wet void will generally be rather less than that for dry voids of the same volume. Flow conditions are therefore usually calculated by the more empirical methods given in the following section, but the above treatment gives a picture of the flow mechanism in a packed column.

## PACKED COLUMNS

Packed columns are used in order to bring about intimate contact between two immiscible fluids—either a liquid and a gas or two liquids. Countercurrent flow is normally employed, with the gas or lighter liquid entering at the bottom and the other fluid entering at the top. An example of a liquid-gas system is an absorption process where a soluble gas is scrubbed from a mixture of gases by means of a liquid, or a distillation column where the more volatile component is transferred from the liquid phase to the vapour phase while the less volatile material is transferred in the opposite direction. Columns are also used for liquid-liquid extraction processes where a solute is transferred from one solvent to another. The principles involved in the design and operation of such columns will be illustrated by consideration of a liquid-gas system in which the liquid flows downwards under the action of gravity. In this chapter the construction of the column and the flow characteristics will be dealt with, whereas the magnitude of the mass transfer coefficients will be discussed in later chapters. In order to obtain a high rate of transfer a packing is employed, which gives a large interfacial area per unit volume of tower, and turbulent conditions at normal rates of flow.

The shell of the tower may be constructed of metal, ceramic material or plastic, or it may be steel with a corrosion-resistant lining. The packing is supported on a grid or screen which must be of an open pattern so as not to offer a high resistance to flow. The liquid is introduced at the top and must be evenly distributed over the whole cross-section. In some cases a number of sprays are arranged at the head of the column, but if the liquid is corrosive or contains small solids in suspension, the sprays are likely to become blocked. In that case, distribution is effected either through a perforated plate, or by allowing the liquid to flow over weirs on a distributing plate. If the ratio of the height to the diameter of the tower is greater than about five to one, bad distribution will result unless redistributing points are introduced for the liquid. It is essential to maintain good distribution, otherwise the effective interfacial area is reduced and, whereas some of the liquid becomes saturated at an early stage, the remaining liquid leaves the column without adequate contact with the gas stream. The largest columns are about 80 feet tall and 30 feet in diameter, whereas laboratory columns are often as small as 1 inch diameter.

## Packings

Packings can be divided into three classes; broken solids, shaped packings and grids. Broken solids are the cheapest form of packing and have been used in sizes from about ½ in. up to 6 in., according to the size of the tower and its operation. They are rarely used now because blockage often occurs if the fluids contain solids in suspension. The smaller the packing the larger is the interfacial area for a given volume of column, and in general the greater the transfer per unit height, but the higher is the pressure drop. It is therefore necessary to balance these two factors against one another for each particular application. The packing should be of uniform size so as to produce a bed of high voidage and low pressure-drop characteristics. Broken solids are occasionally useful because they are obtainable in a wide range of corrosion resistant materials such as brick, stone and coke; they must be sufficiently strong mechanically for degeneration to be inappreciable during operation.

The most commonly used packings in chemical plants are Raschig Rings, Lessing Rings, and Berl Saddles; these are illustrated in Fig. 11.10. They are available in a range of corrosion resistant ceramic materials as well as in metals. A non-porous solid should always be used if there is any risk of crystal formation as the packing dries. Channelling is less marked with shaped packings and the resistance to flow is also smaller; further they give a larger interfacial area per unit volume of tower and the area rendered ineffective because of surface contacts is reduced to a minimum. The great disadvantage is the higher cost particularly with the smaller sizes, because the number of pieces required for a given volume of tower space is inversely proportional to the cube of the linear dimension of the packing. It should be remembered, however, that a smaller tower volume will suffice when the smaller packings are used. The voidage range obtainable with these packings is from about 0·4 to 0·95 (generally $> 0{\cdot}80$) and the performance varies according to the method of packing. Elements larger than 3 in. are often individually stacked so that channelling is reduced to a minimum (Fig. 11.11), but this is a costly process. It is more usual therefore to "dump" the packing in the tower sometimes, when full of liquid. In the laboratory, a number of special packings are often used but these are much too expensive for use in an industrial plant. They include Dixon packings, which are small Lessing Rings made from fine wire gauge, and Stedman packings which are in the form of gauze cones. These packings are very light, and they give high transfer coefficients and low pressure drops by virtue of their small liquid hold-up.

Grid packings have recently been introduced for use in columns up to about three feet across. They are often made in square sections as shown in Fig. 11.12 so that they just fit inside square section columns. They may be made of wood, metal, carbon or ceramic material. This type of packing has a number of advantages: it gives a low pressure drop, the column is readily dismantled and repacked; because of the relatively large spaces in the grids, liquids containing solids in suspension will not cause blockage; very low liquid rates

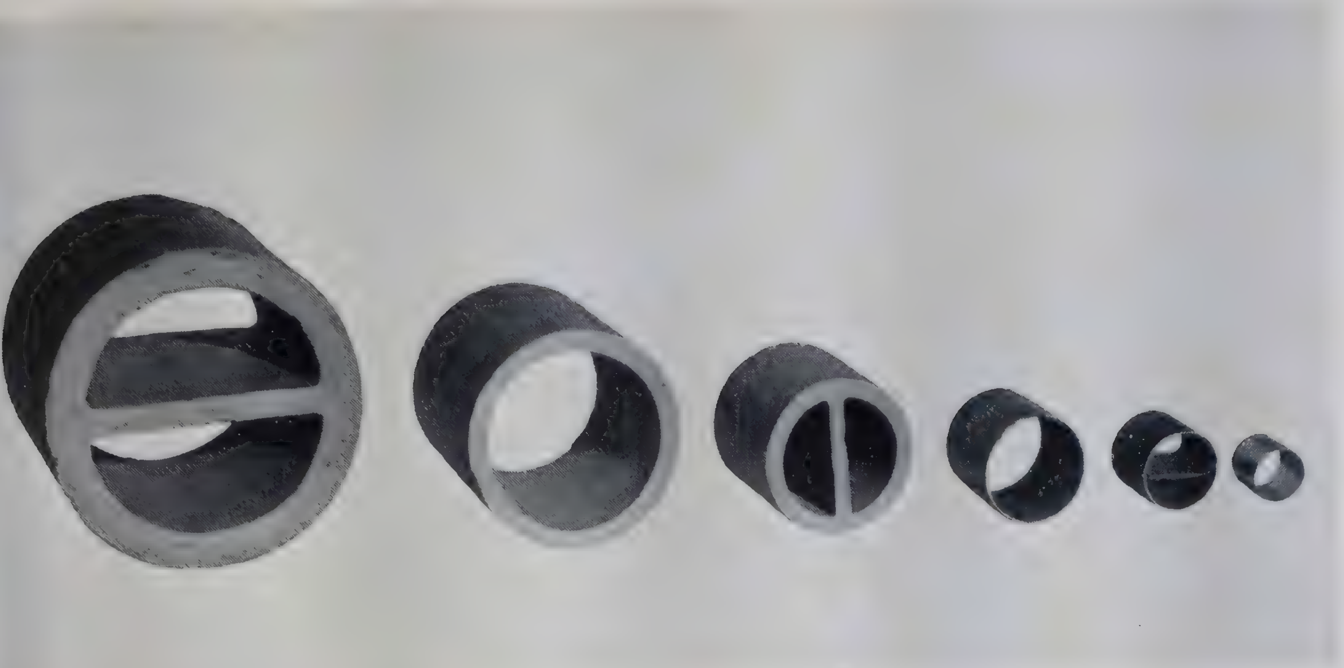


Fig. 11.10*a*. Ceramic and metallic Raschig rings and Lessing rings (made in sizes from 6 in. to $\frac{1}{16}$ in.)

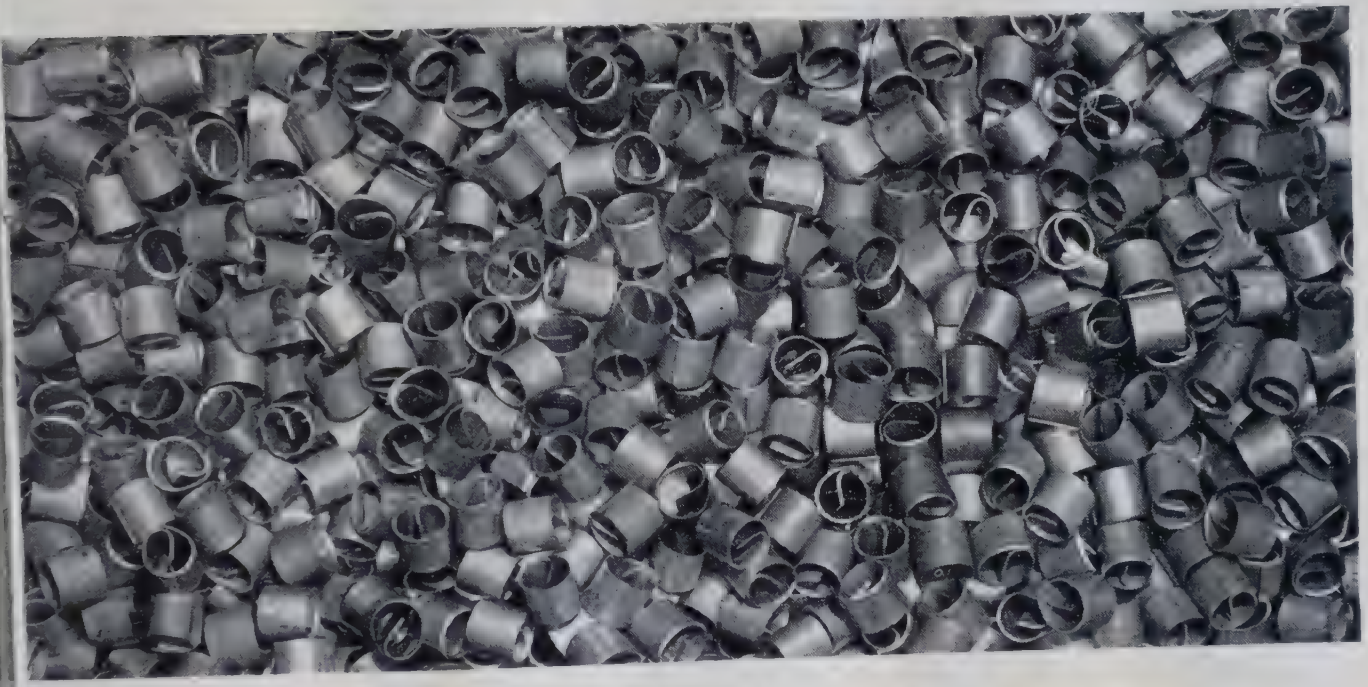


Fig. 11.10*b*. Metallic Lessing rings

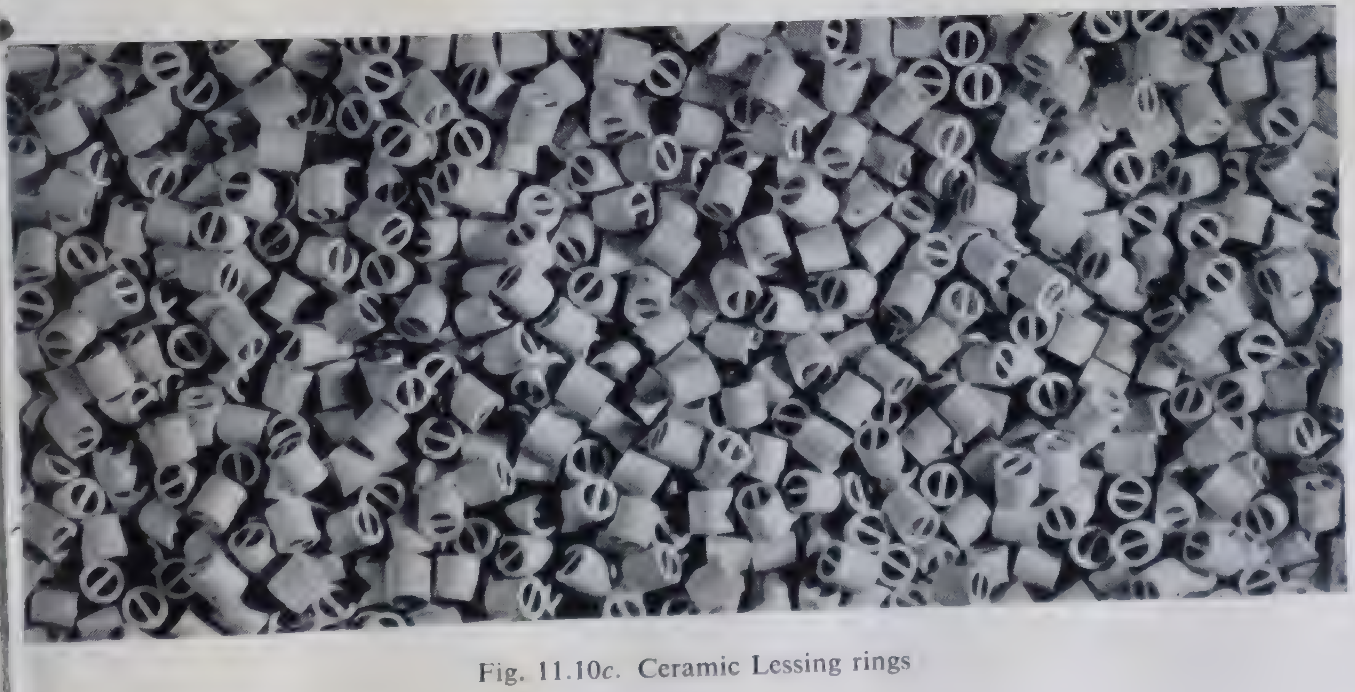


Fig. 11.10*c*. Ceramic Lessing rings

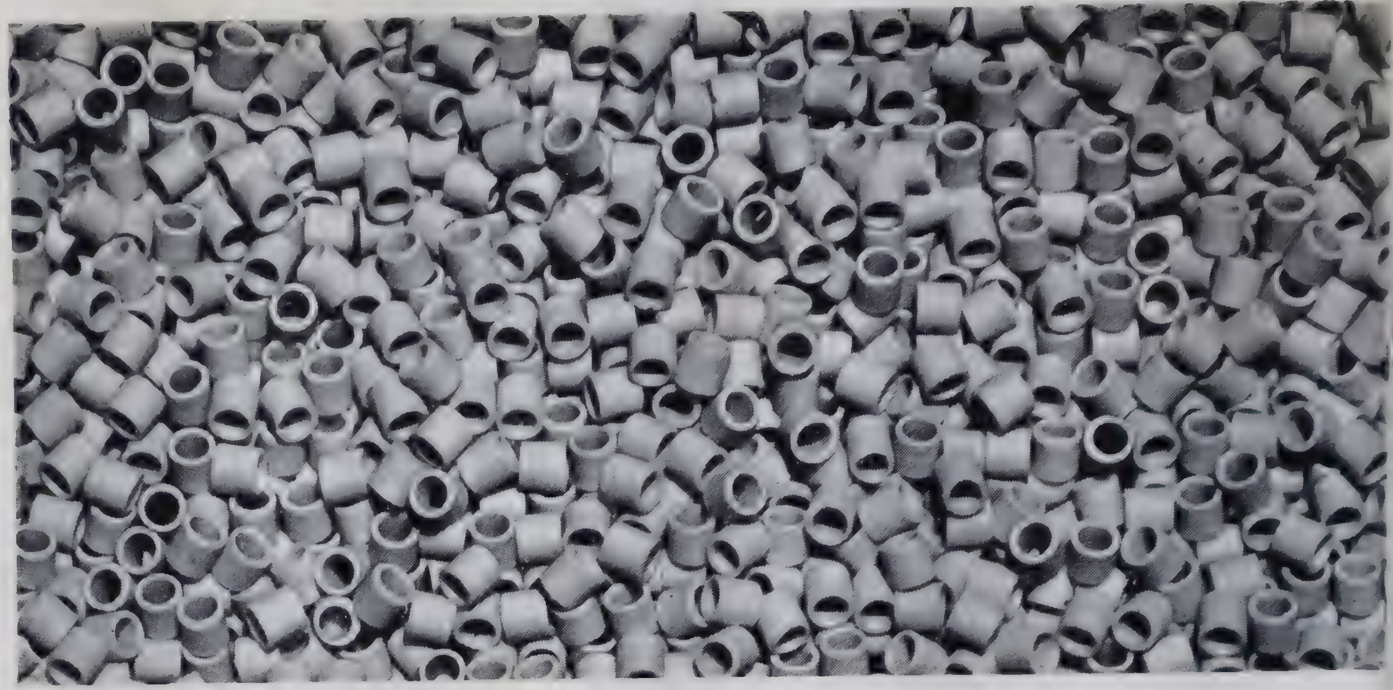


Fig. 11.10*d*. Ceramic Raschig rings

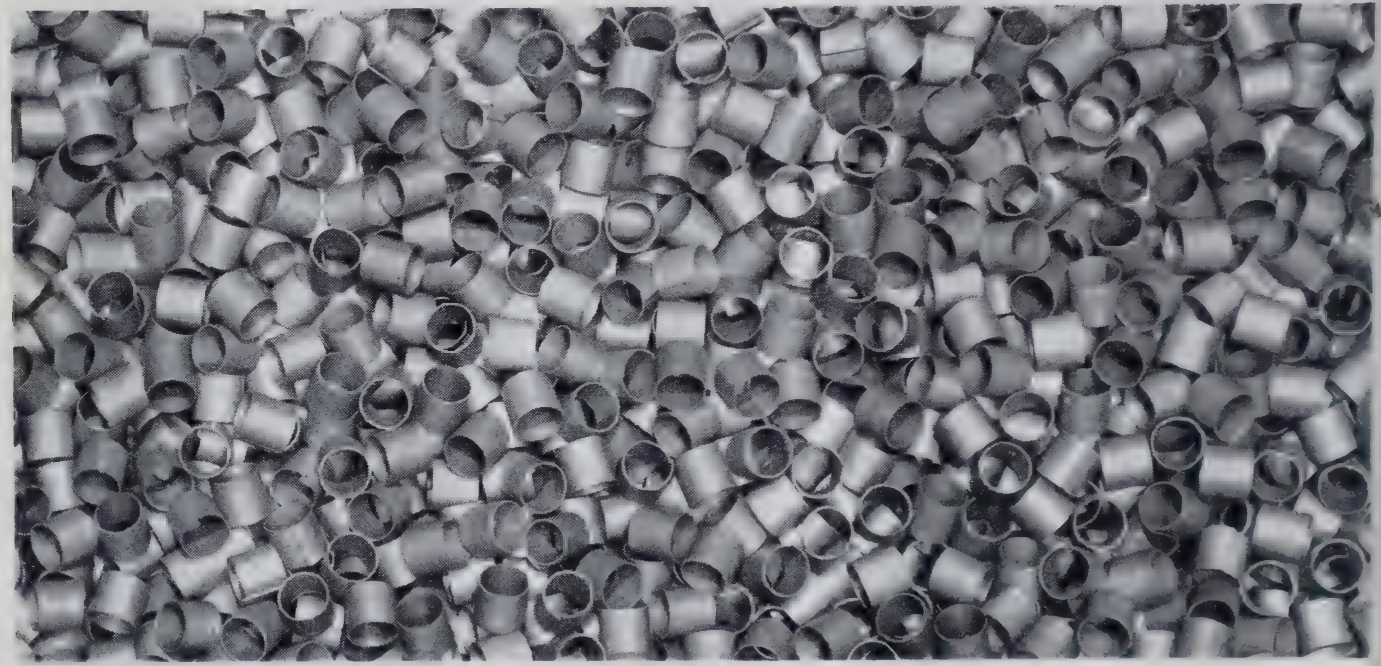


Fig. 11.10*e*. Metallic Raschig rings

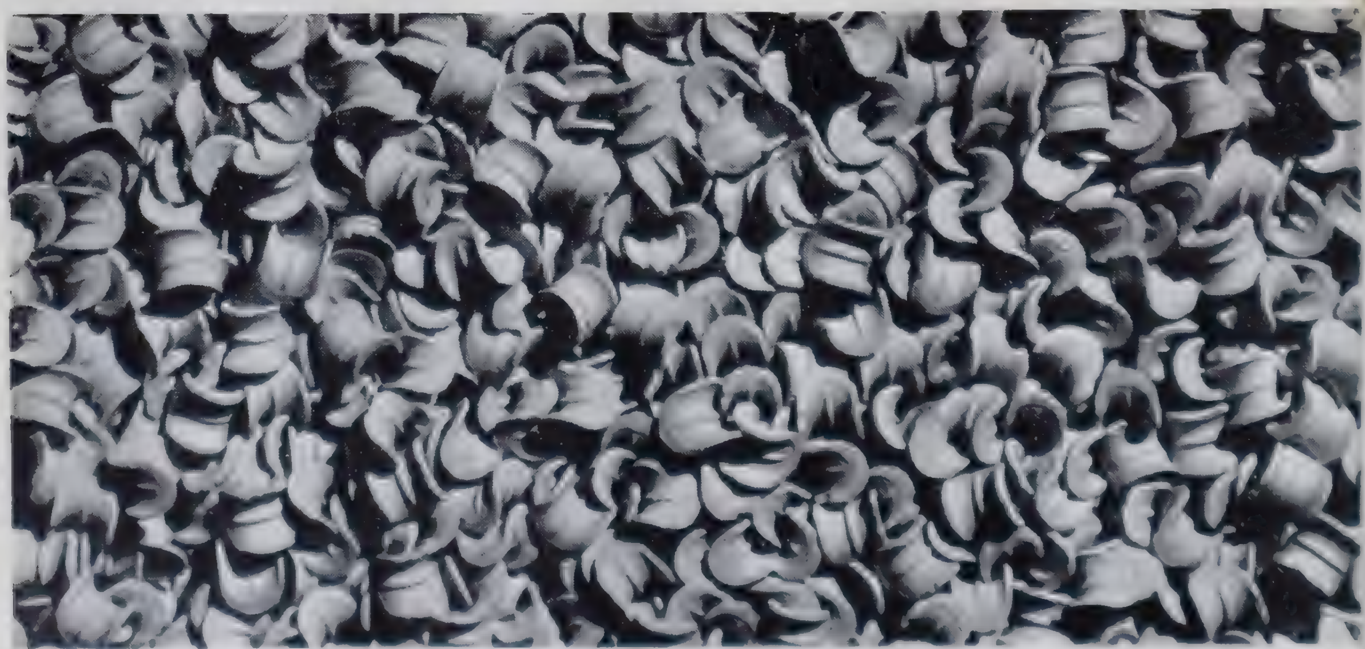


Fig. 11.10*f*. Ceramic Berl saddles

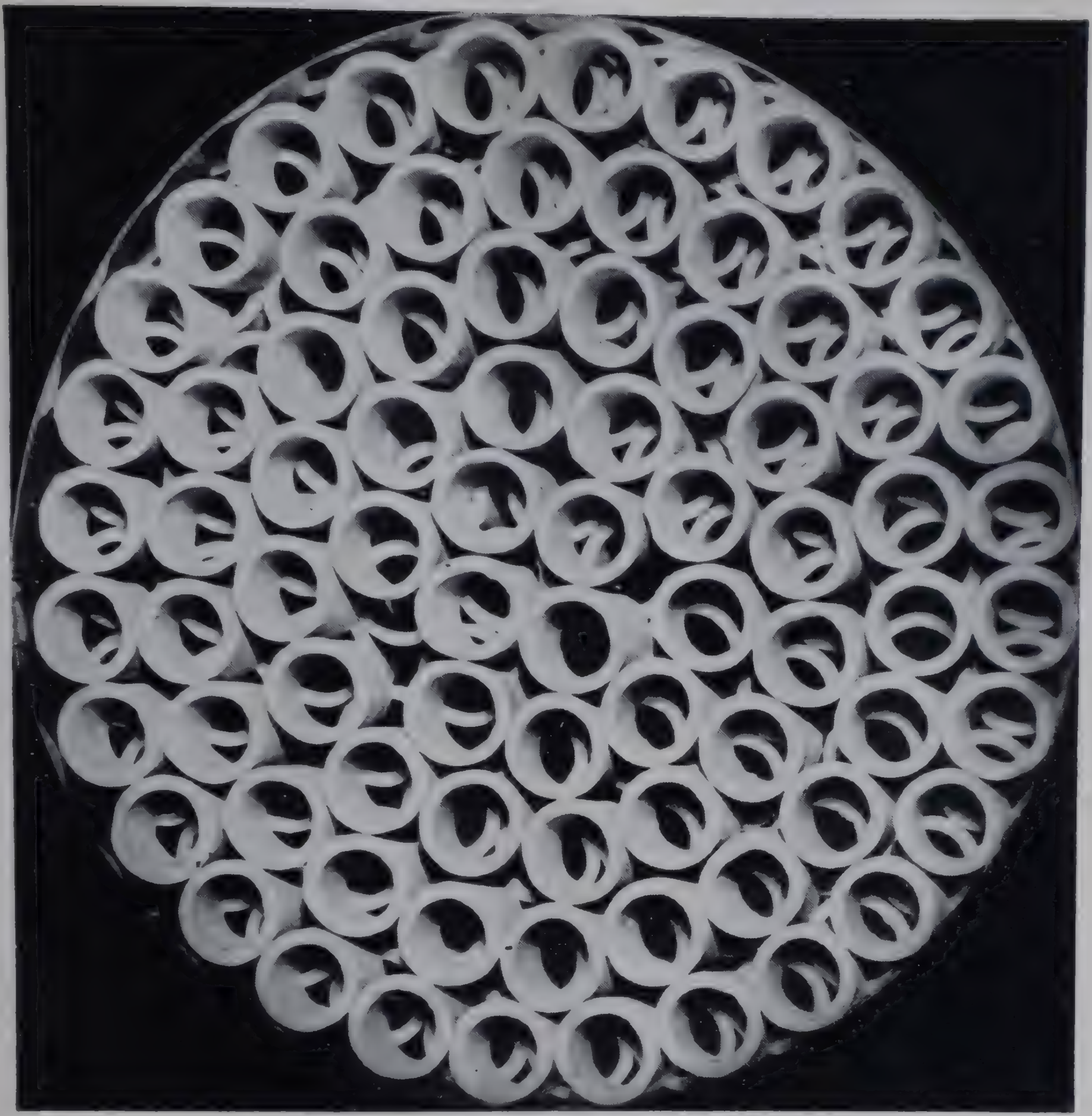


Fig. 11.11. Arrangement of stacked rings

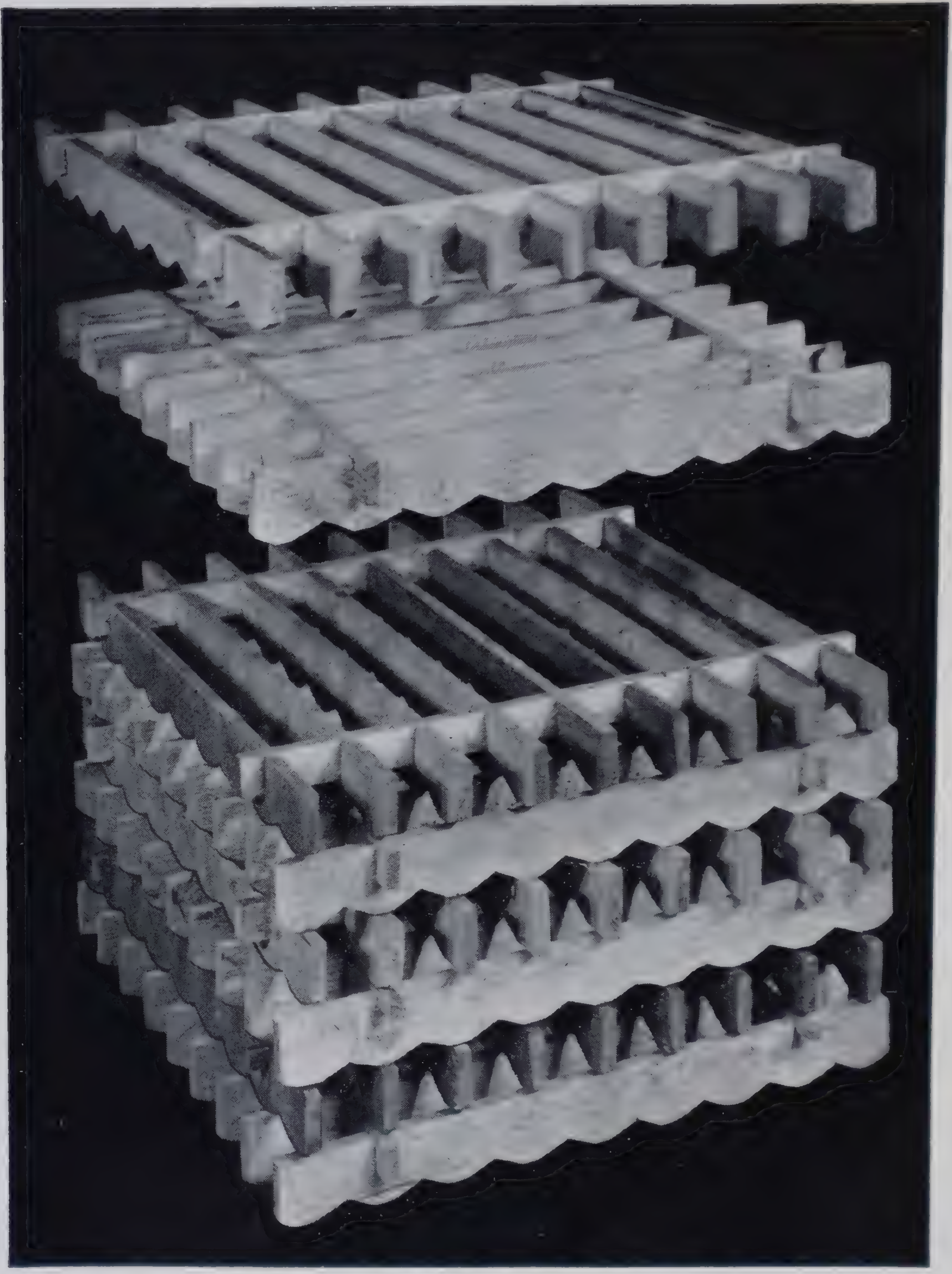


Fig. 11.12. Grid packings

and high gas rates can be employed, with the result that the packing is very suitable where high gas : liquid ratios are required. The most serious disadvantage is that distribution of the liquid is difficult, and at high flow rates liquid cascades from one grid to the next without being broken up into fine droplets. The addition of drip points to the grids is advantageous in this respect.

## Drop in Pressure in Packed Columns

The pressure drop in a column results partly from skin friction and partly from form drag. As there is a direct connection between skin friction and heat and mass transfer coefficients, as indicated in Chapters 8 and 9, it is advantageous that as high a proportion as possible of the frictional resistance should be in the form of skin friction. Chilton and Colburn[(24)] have estimated that not more than 10% of the drag is normally attributable to skin friction.

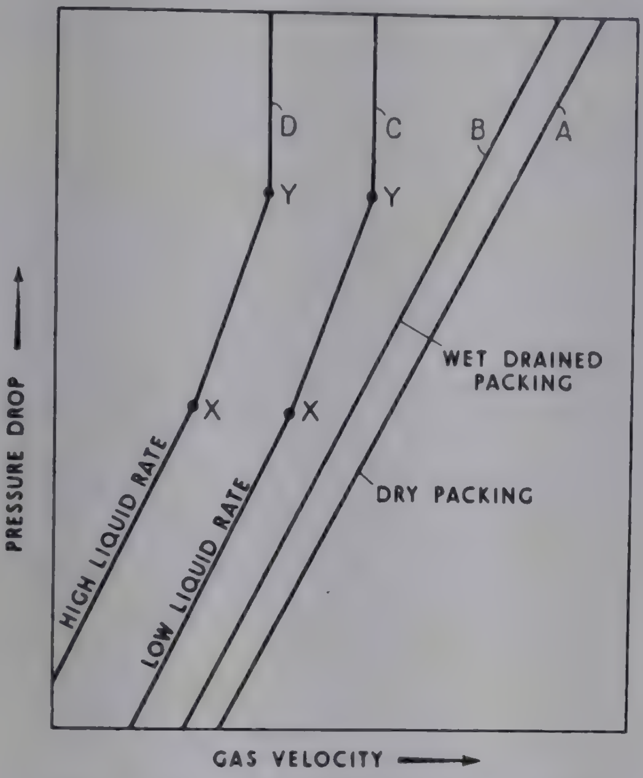


Fig. 11.13. Pressure drops in wet packings

It has been pointed out that it is difficult to obtain reliable information on the flow rates of the gas and liquid from considerations of the proportion of the voids occupied by the two fluids, and it is therefore found more satisfactory to consider how the pressure drop for a given flow rate of gas is modified by the presence of liquid flowing at different rates. As we are concerned here almost exclusively with turbulent flow, the pressure drop for gas flowing through a dry bed will be proportional to its mass rate of flow raised to a power between 1·8 and 2. This is shown by curve A on Fig. 11.13. If the column is flooded with liquid and then allowed to drain and the pressure drop is again measured, it is usually found to be substantially increased (curve B). This is in contradiction to the findings of Wyckoff and Botset, already referred to in connection with their work with sands. If liquid is now allowed to flow countercurrently, the pressure drop is again increased and curve C or D is obtained; this is parallel in the initial stages to curves A and B. If the gas rate is increased beyond a certain point X, it is found that the pressure drop then starts to increase more rapidly, until eventually at point Y the curve rises almost parallel to the pressure axis. The point X is referred to as the loading point, and point Y as the flooding point. For conditions represented by the first section of curve C or D, the drag between the liquid and the gas appears to be small with the liquid merely blocking part of the free void space. At velocities above those corresponding to the loading point, there is an appreciable drag between the phases. Beyond the flooding point the liquid is filling a large proportion of the column and the gas has to bubble through the liquid, so that a very high pressure drop is required to maintain the gas flow. As the gas passes upwards it prevents the drainage of the liquid which then builds up over the top of the packing.

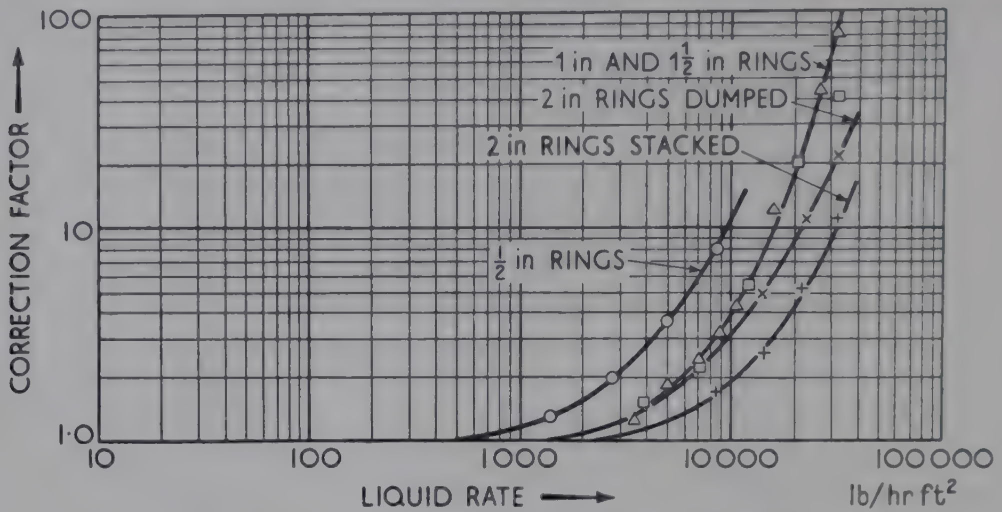


Fig. 11.14. Correction factor for liquid flow (Raschig rings)

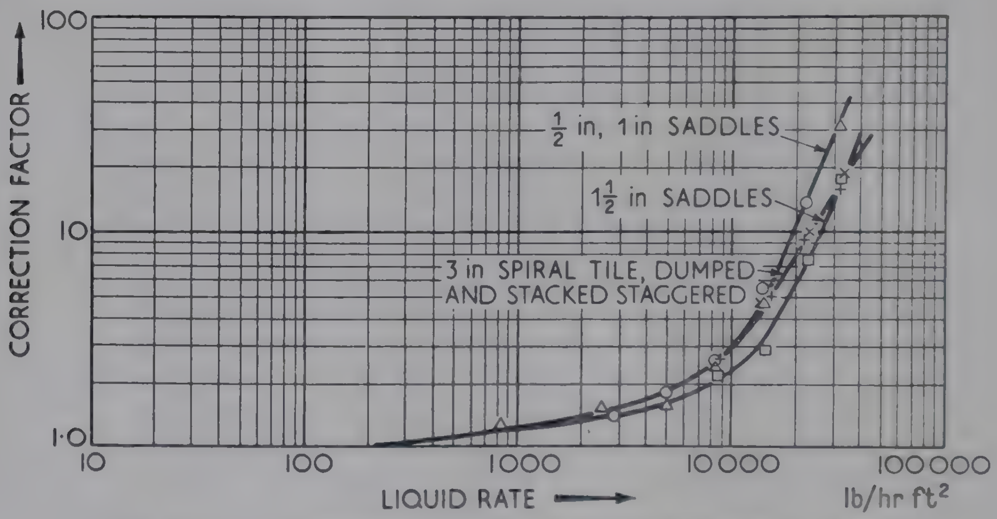


Fig. 11.15. Correction factor for liquid flow (Berl saddles)

The method of calculation of the difference in pressure across the column consists of using one of the equations already given (e.g. 11.14 or 11.17) for the pressure drop due to the gas, and then multiplying by an experimentally determined correction factor to allow for the liquid flow. If the equation of CHILTON and COLBURN or of ROSE is employed, it is necessary to include a factor to take account of the particle shape and the voidage of the bed, since their correlation was based on the average velocity and a mean diameter of the particles. Whereas this was moderately satisfactory for beds composed of ordinary granular material, it is no longer applicable where the voidage reaches the very high values that are obtained in packed columns. A further factor is also used to take account of the wall effect. The various correction factors, for water as the aqueous phase, are expressed graphically in Figs. 11.14 to 11.17 which are taken from Sherwood and Pigford.[(5)]

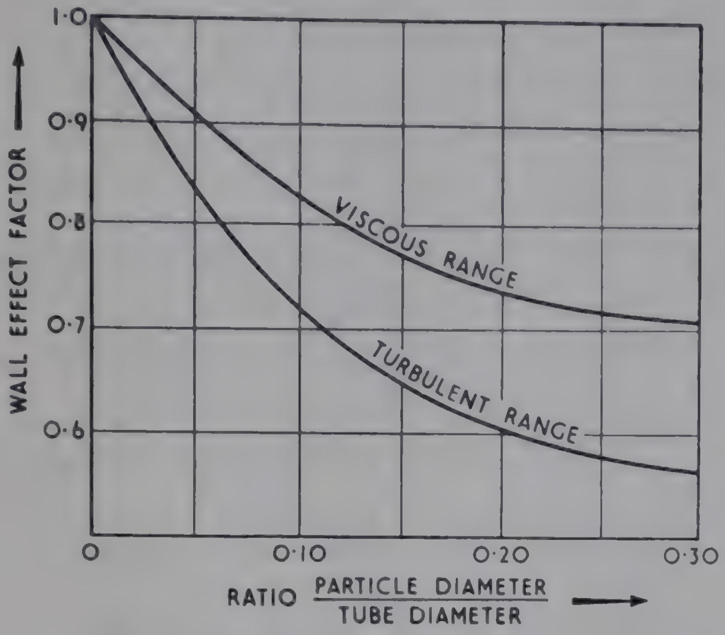


Fig. 11.16. Correction factor for wall effect

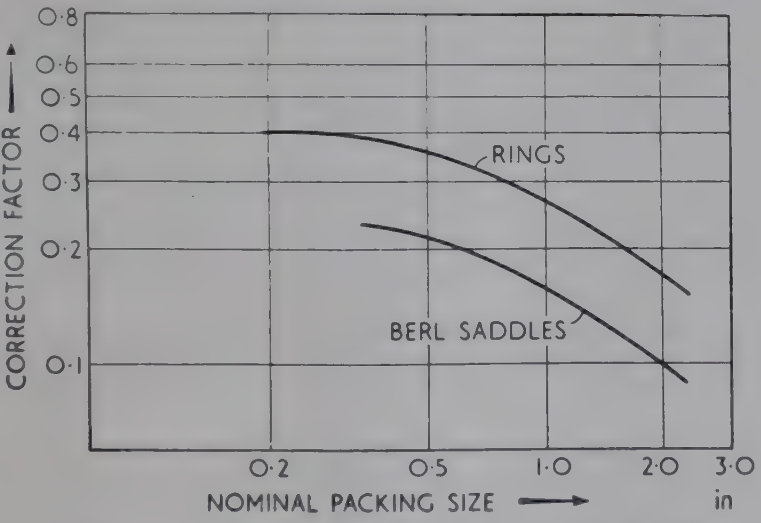


Fig. 11.17. Correction factor for shape of packing

In Fig. 11.14 is given the factor by which the pressure drop for a dry packing must be multiplied, in order to give the actual pressure drop for any rate of flow of water over Raschig rings. These curves relate to relatively large packings for which the pressure drop for the wet drained packing is similar to that for the dry condition. With small packings, the resistance in the wet drained condition will be appreciably greater, but there are comparatively few data available on this point. Rose and Young[(62)] give the following equation from their experimental work with Raschig rings.

$$\Delta P_w = \Delta P_d \left(1 + \frac{0{\cdot}33}{d_n}\right) \qquad \ldots .(11.24)$$

where $\Delta P_w$ is the pressure drop across the wet drained column,
$\Delta P_d$ is the pressure drop across the dry column and
$d_n$ is the nominal size of the Raschig rings in *centimetres.*

The curve for the effect of liquid rate with Berl saddles is given in Fig 11.15, but no corresponding figures are available for the relation between $\Delta P_w$ and $\Delta P_d$. The slopes of the curves in Figs 11.14 and 11.15 increase rapidly at high liquid rates as the flooding condition is reached.

The correction factor for the wall effect is shown in Fig. 11.16. This is based on the work of FURNAS(23). Separate curves are obtained for streamline and turbulent flow but we are concerned only with the turbulent region.

Correction factors for use with CHILTON and COLBURN's and ROSE's expressions for pressure drop are given in Fig. 11.17. The data on voidage and surface per unit volume of packed space given in Table 11.1 can be used for the calculation of pressure drops using CARMAN's equation (11.14).

TABLE 11.1. *Particulars of Hy-Contact Tower Packings* (approximate values)*

| Packing | Size in. | Wall thickness in. | Number per cu ft | Weight lb per cu ft | Contact surface $a$, sq ft per cu ft | Free space, $100e$ per cent |
|---|---|---|---|---|---|---|
| Lessing Rings Stoneware | $4 \times 4$ | 0·44 | 21 | 45 | 21 | 70 |
| | $2 \times 2$ | 0·24 | 160 | 43 | 34 | 69 |
| | $1 \times 1$ | 0·13 | 1,300 | 49 | 68 | 68 |
| | $\frac{3}{4} \times \frac{3}{4}$ | 0·09 | 2,850 | 46 | 85 | 72 |
| | $\frac{1}{2} \times \frac{1}{2}$ | 0·07 | 10,300 | 52 | 130 | 64 |
| | $\frac{3}{8} \times \frac{3}{8}$ | 0·05 | 23,500 | 47 | 175 | 69 |
| | $\frac{1}{4} \times \frac{1}{4}$ | 0·03 | 89,000 | 49 | 294 | 65 |
| | $\frac{3}{16} \times \frac{3}{16}$ | 0·025 | 208,000 | 56 | 389 | 63 |
| Lessing Rings Mild Steel | $3 \times 3$ | 0·048 | 50 | 25 | 26 | 95 |
| | $2 \times 2$ | 0·048 | 160 | 36 | 38 | 93 |
| | $1 \times 1$ | 0·028 | 1,350 | 43 | 80 | 91 |
| | $\frac{3}{4} \times \frac{3}{4}$ | 0·022 | 2,850 | 41 | 95 | 92 |
| | $\frac{1}{2} \times \frac{1}{2}$ | 0·018 | 10,500 | 55 | 158 | 89 |
| | $\frac{3}{8} \times \frac{3}{8}$ | 0·015 | 20,000 | 46 | 170 | 90 |
| | $\frac{1}{4} \times \frac{1}{4}$ | 0·015 | 68,000 | 70 | 263 | 85 |
| | $\frac{1}{8} \times \frac{1}{8}$ | 0·012 | 400,000 | 88 | 404 | 82 |
| Raschig Rings Stoneware | $4 \times 4$ | 0·44 | 23 | 37 | 16 | 74 |
| | $2 \times 2$ | 0·25 | 170 | 39 | 29 | 73 |
| | $1 \times 1$ | 0·14 | 1,350 | 43 | 58 | 71 |
| | $\frac{3}{4} \times \frac{3}{4}$ | 0·09 | 3,000 | 39 | 73 | 77 |
| | $\frac{1}{2} \times \frac{1}{2}$ | 0·07 | 10,500 | 42 | 112 | 71 |
| | $\frac{3}{8} \times \frac{3}{8}$ | 0·05 | 26,000 | 52 | 157 | 73 |
| | $\frac{1}{4} \times \frac{1}{4}$ | 0·03 | 90,000 | 40 | 242 | 72 |
| | $\frac{1}{8} \times \frac{1}{8}$ | 0·02 | 580,000 | 42 | 385 | 72 |
| Raschig Rings Mild Steel | $3 \times 3$ | 0·064 | 54 | 28 | 22 | 94 |
| | $2 \times 2$ | 0·048 | 170 | 29 | 31 | 94 |
| | $1 \times 1$ | 0·028 | 1,400 | 35 | 63 | 93 |
| | $\frac{3}{4} \times \frac{3}{4}$ | 0·022 | 3,300 | 36 | 84 | 93 |
| | $\frac{1}{2} \times \frac{1}{2}$ | 0·018 | 11,100 | 44 | 127 | 91 |
| Raschig Rings Carbon | $3 \times 3$ | 0·312 | 49 | 23 | 19 | 78 |
| | $2 \times 2$ | 0·250 | 166 | 27 | 29 | 74 |
| | $1 \times 1$ | 0·125 | 1,325 | 27 | 57 | 74 |
| | $\frac{3}{4} \times \frac{3}{4}$ | 0·125 | 3,140 | 34 | 75 | 67 |
| | $\frac{1}{2} \times \frac{1}{2}$ | 0·063 | 10,600 | 27 | 114 | 74 |
| | $\frac{1}{4} \times \frac{1}{4}$ | 0·063 | 85,000 | 46 | 212 | 55 |
| Berl Saddles Stoneware | 2 | — | 270 | 40 | 35 | 77 |
| | 1 | — | 2,250 | 45 | 74 | 70 |
| | $\frac{3}{4}$ | — | 4,800 | 48 | 95 | 69 |
| | $\frac{1}{2}$ | — | 16,000 | 55 | 142 | 66 |
| | $\frac{3}{8}$ | — | 40,000 | 55 | 190 | 65 |
| | $\frac{1}{4}$ | — | 145,000 | 56 | 350 | 65 |

* Reproduced by courtesy of the Hydronyl Syndicate Ltd.

## Flow Rates

The flow rates should be adjusted so that the pressure drop is as low as possible, consistent with the tower carrying out its desired functions, since the power required to force the gas through the column is proportional to the product of the gas rate and the drop in pressure. It will be seen in Chapter 19, that the ratio of gas flow to liquid flow must not exceed a certain value if the column is to effect the required degree of absorption, and the lower the ratio the smaller is the required height of the tower.

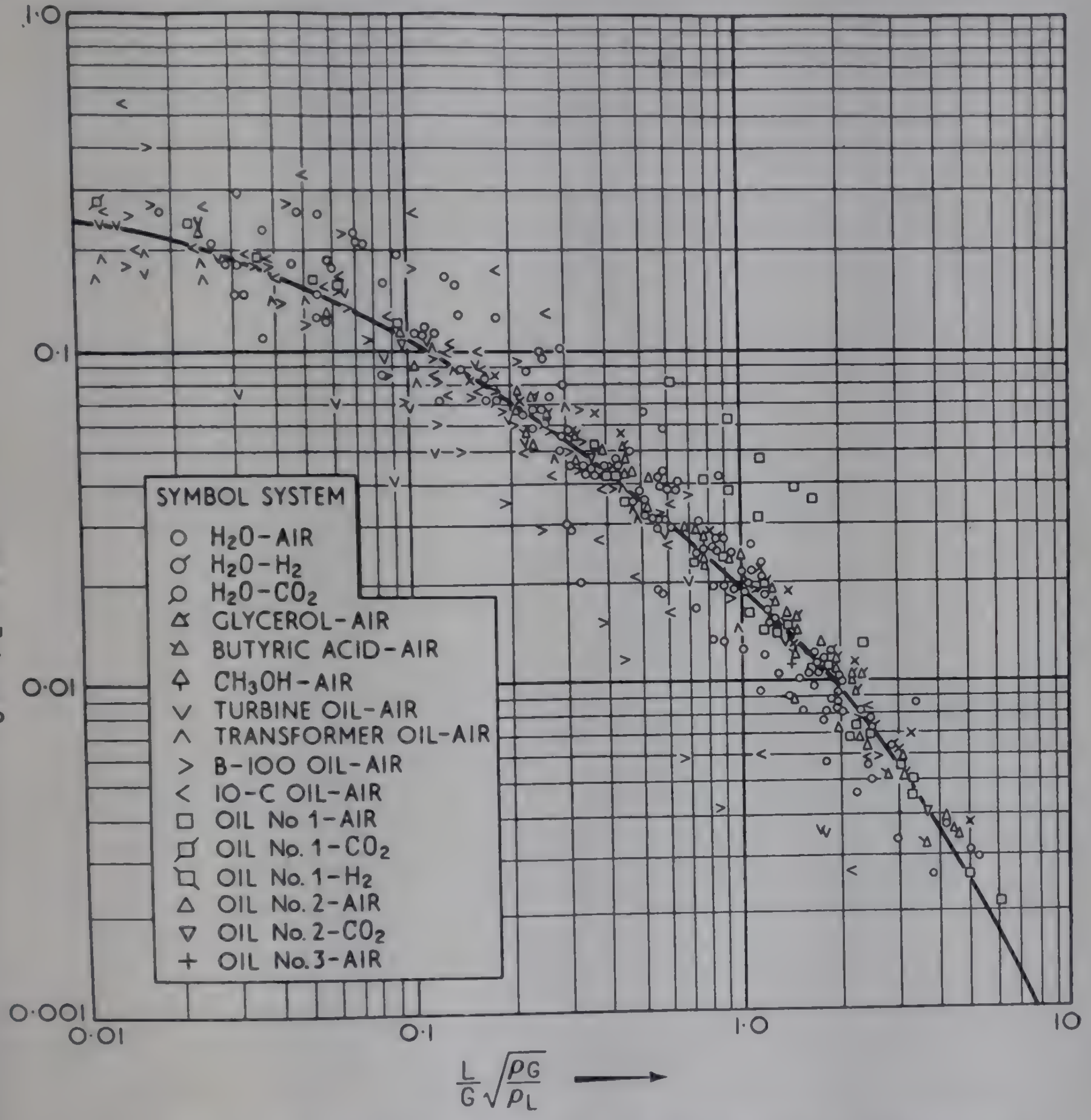


Fig. 11.18. Generalised correlation for flooding rates in packed towers

The liquid rate must be sufficient for the packing to be thoroughly wetted. Morris and Jackson[(6)] suggest that the required flow of liquid is directly proportional to the periphery of the packing elements, the periphery being defined as the surface of the packing divided by a characteristic length. Since the periphery per unit cross-section of column is equal to the surface of packing per unit volume, the irrigation rate can be expressed as

$$\frac{\text{liquid rate (ft}^3\text{/hr-ft}^2\text{)}}{\text{Surface area of packing per unit volume of column (ft}^2\text{/ft}^3\text{)}}$$

The minimum irrigation rate should be taken as $0{\cdot}85$ ft$^3$/hr-ft for rings between 1 in. and 3 in. diameter and grids of pitch less than 2 in. For larger materials, an irrigation rate of about $1{\cdot}3$ ft$^3$/hr-ft is required. Other workers quote rather lower figures. If it is impossible to obtain this liquid rate by normal operation, the column may be flooded at intervals. Alternatively, part of the liquid may be recirculated but the advantages of countercurrent flow are then lost.

The maximum rate of flow is determined by the onset of flooding. The flooding point, as already mentioned, is characterized by a rapid increase in the pressure drop but it can also be detected visually in a glass column. The flooding point, as detected visually, usually occurs at a slightly lower flow rate than that indicated by the curve (Fig. 11.13). It represents a condition where the hold-up of liquid in the column increases rapidly and, in order to avoid reaching this condition, liquid rates should not generally exceed about 60% of the flooding velocity. The flooding rate is a function of the gas rate and no accurate correlation of the conditions at which flooding occurs has been developed. The best available information is that given by LOBO *et al.*(36) and is based on an earlier correlation by SHERWOOD *et al.*(33) In Fig. 11.18, a curve is given which represents the conditions at which flooding commences for a wide range of packings and gas rates. A logarithmic scale is used and the groups which are plotted are

$$\frac{u_G{}^2 a}{g e^3}\left(\frac{\rho_G}{\rho_L}\right)\left(\frac{\mu_L}{\mu_w}\right)^{0\cdot2} \quad \text{and} \quad \frac{L}{G}\sqrt{\frac{\rho_G}{\rho_L}}$$

where $u_G$ is the velocity of the gas, calculated over the whole cross-section of the bed,

$a$ is the surface of the packing per unit volume of bed, $S(1 - e)$,

$g$ is the acceleration due to gravity,

$L$ is the mass rate of flow of the liquid,

$G$ is the mass rate of flow of the gas,

and $\mu_w$ is the viscosity of water at 20°C (approximately 1 centipoise)

Suffix $G$ refers to the gas and suffix $L$ to the liquid.

The area inside the curve represents possible conditions of operation. In the above expressions, the ratios $\frac{\rho_G}{\rho_L}$ and $\frac{\mu_L}{\mu_w}$ have been introduced so that the relationship can be applied for a wide range of liquids and gases.

### Flow of Two Liquids

The work which has been carried out with two liquids is less extensive than that for gas-liquid systems. Some of the most recent work has been done by PRATT and his co-workers(60, 61) and they find that the characteristics of the two systems are similar, in that three regimes of flow are obtained, separated by the loading point and the flooding point. A correlation of flooding rates is given by the following equation.

$$1 + 0{\cdot}835 \left(\frac{\rho_d}{\rho_c}\right)^{\frac{1}{4}} \left(\frac{u_d}{u_c}\right)^{\frac{1}{2}} = Z\left[\frac{u_c{}^2 a}{g e^3}\left(\frac{\rho_c}{\Delta\rho}\right)\left(\frac{\sigma}{\sigma_T}\right)^{\frac{1}{4}}\right]^{-\frac{1}{4}} \qquad \ldots.(11.25)$$

Here $a$ is the surface per unit volume of tower, $S(1 - e)$,

$\sigma$ is the interfacial tension,

$\sigma_T$ is the interfacial tension (water-air, 15°C) [73 dynes/cm.],

$\Delta\rho = |(\rho_c - \rho_d)|$,

$Z = 0{\cdot}52$ for Raschig rings,

$0{\cdot}61$ for Lessing rings and

$0{\cdot}67$ for Berl saddles

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## CHAPTER 12

# *Filtration*

## THEORY OF FILTRATION

FILTRATION is the process whereby a solid is separated from a liquid or gas by means of a porous medium which retains the solid but allows the fluid to pass. In general, the pores of the medium will be of a tortuous shape and larger than the particles which are to be removed, and the filter will work efficiently only after an initial deposit has been trapped in the medium. In the chemical laboratory, filtration is often carried out in a form of Buchner funnel, and the liquid is sucked through the thin layer of particles using a source of vacuum: in even simpler cases the suspension is poured into a conical funnel fitted with a filter paper. In the industrial equivalent of such an operation we are faced with the difficulties involved in the mechanical handling of much larger quantities of suspension and solids. We shall have to allow a thicker layer of solids to form and, in order to achieve a high rate of passage of liquid through the solids, higher pressures will be needed. Alternatively, it will be necessary to provide a far greater area. A typical filtration operation is illustrated in Fig. 12.1, which shows the filter medium, in this case a cloth, its support and the layer of solids, or filter cake, which has already formed.

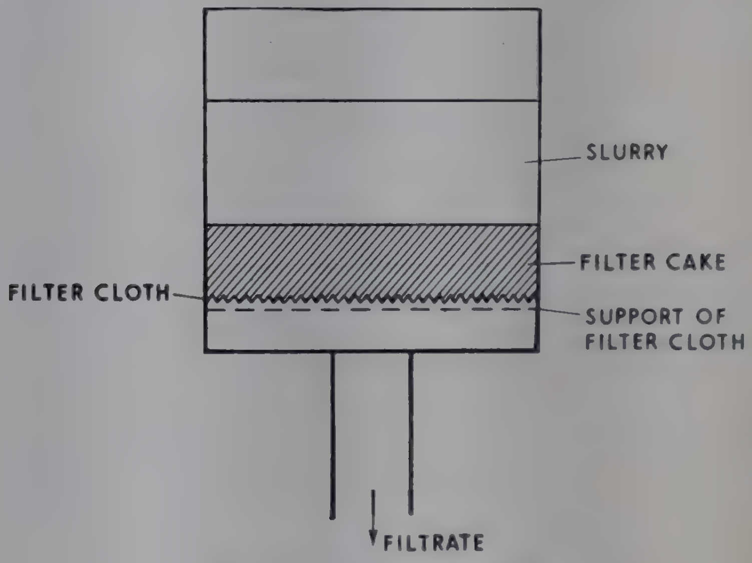


Fig. 12.1. A simple filter

The conditions under which filtration is carried out are very varied and the choice of the most suitable type of equipment will depend on a large number of factors including:

(1) The properties of the fluid, particularly its viscosity, density and corrosive properties.

(2) The nature of the solid—its particle size and shape, size distribution, and packing characteristics.
(3) The concentration of solids in suspension.
(4) The quantity of material to be handled, and its value.
(5) Whether the valuable product is the solid, the fluid, or both.
(6) Whether it is necessary to wash the filtered solids.
(7) Whether very slight contamination caused by contact of the suspension or filtrate with the various components of the equipment is detrimental to the product.

Whatever type of equipment is used, a filter cake gradually builds up on the medium and the resistance to flow increases progressively throughout the operation. The flow problem can be regarded in two parts: first the flow of fluid through the filter medium and the initial layer of solids, and secondly the flow through a bed formed by the particles which are being continuously deposited. It would be expected that the relations already developed in Chapter 11 would be applicable here.

The most important factors on which the rate of filtration depends will then be:

(1) The drop in pressure from the feed to the far side of the filter medium.
(2) The area of the filtering surface.
(3) The viscosity of the filtrate.
(4) The resistance of the filter cake.
(5) The resistance of the filter medium and initial layers of cake.

The effects of these variables will now be investigated, and relations developed for the rate of filtration obtained under various conditions. Consideration will be given to the desirable features to be incorporated in filtration equipment for any given purpose, and some of the more common types will be discussed.

### Flow through the Filter Cake

In Chapter 11, equations were obtained for the calculation of the rate of flow of a fluid through a bed of granular material, and these will now be applied to the flow of filtrate through the filter cake. Some differences in general behaviour may be expected, however, because the cases so far considered relate to uniform fixed beds, whereas in filtration the bed is steadily growing in thickness. Thus if the filtration pressure is constant, the rate of flow will progressively diminish, whereas if the flow rate is to be maintained constant, the pressure must be gradually increased.

Because the particles forming the filter cake are normally small and the rate of flow of filtrate is low, streamline conditions are almost invariably obtained, and therefore at any instant in the process, equation 11.7 should be applicable.

This may be written in the form

$$\frac{1}{A}\frac{dV}{dt} = \frac{e^3}{5(1-e)^2S^2}\frac{\Delta P}{\mu l} \qquad \text{....(12.1)}$$

In the derivation of this equation it was assumed that the cake was uniform and that the voidage was constant throughout. In some cases, however, the porosity will depend both on the rate of deposition and on the nature of the surface on which the particles are deposited. The initial stages in the formation of the cake are therefore of great importance for the following reasons:

(1) For any filtration pressure, the rate of flow is greatest at the beginning of the process since the resistance is then a minimum.

(2) High initial rates of filtration may result in plugging of the pores of the filter cloth and cause a very high resistance to flow.

(3) The orientation of the particles in the initial layers can appreciably influence the structure of the whole filter cake.

Filter cakes can be divided into two classes, incompressible cakes and compressible cakes. In the case of an incompressible cake, the resistance to flow of a given volume of cake is not appreciably affected either by the pressure difference across the cake or by the rate of deposition of material. On the other hand, with a compressible cake increase of the pressure difference or of the rate of flow causes the formation of a denser cake with a higher resistance. With very compressible cakes, over certain ranges, increase of the pressure difference may actually decrease the rate of filtration. For the present we will confine our attention to incompressible cakes in which $e$ in equation 12.1 can be taken as constant. The quantity $\frac{e^3}{5(1-e)^2S^2}$ is then a property of the particles forming the cake and should be constant for a given material.

We can therefore write,

$$\frac{1}{A}\frac{dV}{dt} = \frac{\Delta P}{r\mu l} \qquad \text{....(12.2)}$$

where

$$r = \frac{5(1-e)^2S^2}{e^3} \qquad \text{....(12.3)}$$

$\Delta P$ is taken here as the fall in pressure and is therefore positive.

Equation 12.2 is the basic filtration equation. $r$ is termed the specific resistance and is numerically equal to the pressure difference required to produce a unit rate of flow of filtrate of unit viscosity through a unit cube of the cake. Its dimensions are $\mathbf{L}^{-2}$.

***Relation between Thickness of Cake and Volume of Filtrate.*** In equation 12.2, the variables $l$ and $V$ are connected, and the relation between them can be obtained as follows.

Mass of solids in filter cake $= (1-e)Al\rho_s$

(where $\rho_s$ is the density of the solids)

Mass of liquid retained in the filter cake $= eAl\rho$

(where $\rho$ is the density of the filtrate)

Then if $J$ is the ratio of the mass fraction of solids in the original suspension,

$$\frac{J}{1-J} = \frac{(1-e)Al\rho_s}{(V+eAl)\rho} = \frac{\text{Mass of solids in filter cake}}{\text{mass of filtrate + mass of liquid retained in cake}}$$

i.e. $(1-J)(1-e)Al\rho_s = JV\rho + AeJl\rho$

so that
$$l = \frac{JV\rho}{A\{(1-J)(1-e)\rho_s - Je\rho\}} \qquad \ldots.(12.4)$$

Writing
$$v = \frac{J\rho}{(1-J)(1-e)\rho_s - Je\rho} \qquad \ldots.(12.5)$$

the volume of cake deposited by the passage of unit volume of filtrate,

$$l = \frac{vV}{A} \qquad \ldots.(12.6)$$

Substituting in equation (12.2),

$$\frac{dV}{dt} = \frac{A^2\Delta P}{r\mu Vv}. \qquad \ldots.(12.7)$$

If the resistance to flow is initially zero and a volume $V$ of filtrate passes in time $t$, the above equation can be integrated provided that some further relation connecting $\Delta P$, $V$ and $t$ is given. Two important cases will be considered.

***Filtration Equations.*** *For a filtration at constant rate,*

$$\frac{dV}{dt} = \frac{V}{t} = \text{constant},$$

so that
$$\frac{V}{t} = \frac{A^2\Delta P}{r\mu Vv} \qquad \ldots.(12.8a)$$

i.e.
$$\frac{t}{V} = \frac{r\mu v}{A^2\Delta P}V \qquad \ldots.(12.8b)$$

and $\Delta P$ is directly proportional to $V$.

*For a filtration at constant pressure,*

$$\frac{V^2}{2} = \frac{A^2\Delta Pt}{r\mu v} \qquad \ldots.(12.9a)$$

i.e.
$$\frac{t}{V} = \frac{r\mu v}{2A^2\Delta P}V \qquad \ldots.(12.9b)$$

Thus for a constant pressure filtration, there is a linear relation between $V^2$ and $t$ or between $\frac{t}{V}$ and $V$.

In equations 12.9$a$ and 12.9$b$, it is assumed that filtration is carried out at constant pressure throughout, i.e. that $V = 0$ when $t = 0$. In most cases, however, $\Delta P$ is gradually increased at the beginning of the operation, so that the pressure is built up to its final value in a time $t_1$ during which a volume $V_1$ of filtrate passes. Then on integration of equation 12.7,

$$\tfrac{1}{2}(V^2 - V_1^2) = \frac{A^2 \Delta P}{r \mu v}(t - t_1) \qquad \ldots.(12.10a)$$

Thus
$$(V - V_1)(V - V_1 + 2V_1) = \frac{2A^2 \Delta P}{r \mu v}(t - t_1)$$

i.e.
$$\frac{t - t_1}{V - V_1} = \frac{r \mu v}{2A^2 \Delta P}(V - V_1) + \frac{r \mu v V_1}{A^2 \Delta P} \qquad \ldots.(12.10b)$$

Thus there is a linear relation between $V^2$ and $t$ and between $\dfrac{t - t_1}{V - V_1}$ and $V - V_1$. Here $t - t_1$ represents the time of the constant pressure filtration and $V - V_1$ the corresponding volume of filtrate obtained.

RUTH(26, 27, 30, 40) has made measurements on the flow in a filter cake and has concluded that the resistance is somewhat greater than that indicated by equation 12.1; he assumes that part of the pore space is rendered ineffective for the flow of filtrate because of the adsorption of ions on the surface of the particles. This is not borne out by GRACE(65) or by HOFFING and LOCKHART(55) who determined the relation between flow rate and pressure difference, both by means of permeability tests on a fixed bed and by filtration tests using suspensions of quartz and diatomaceous earth.

### The Filter Medium

The function of the filter medium is generally to act as a support for the filter cake, while the initial layers of cake provide the true filter. It is therefore important that the commencement of the operation should be carried out with great care. In the case of very thick materials, such as felt or flannel, some of the fine particles pass through the bed and are trapped in the medium itself. The filter medium should be mechanically strong, resistant to the corrosive action of the fluid, and offer as little resistance as possible to the flow of filtrate. A relatively coarse material is often used therefore and clear filtrate is not obtained until the initial layers of cake are formed; this filtrate should then be recycled.

The most important filter media are:—

(1) Woven materials, including wool, cotton, linen, silk, glass, plastic (e.g. nylon) and metal.
(2) Perforated metal sheeting.
(3) Granular materials, including gravel, sand, asbestos, charcoal and kieselguhr.
(4) Porous solids.

Woven materials are most commonly used but granular materials and porous solids are particularly useful for the filtration of very corrosive liquids. We shall confine our attention to flow through woven filter cloths. For these materials the type of weave is very important as it often affects the adherence of the cake to the cloth.

***Flow of Liquid through the Filter Cloth.*** Experiments[(32)] show that the rate of flow of the fluid through the filter cloth is directly proportional to the pressure difference at low rates of flow, but that it becomes proportional to the square root of the pressure difference at higher rates. In the former case streamline conditions exist and in the latter case the flow rate has become sufficiently high for turbulence to set in; such high rates are not normally encountered in filtration.

At low rates of flow, some deviations from the linear relation are found with new cloths or cloths that are not well supported. The rate of flow is then proportional to the pressure raised to a power greater than unity. This deviation is attributable to stretching, and the cloth does not subsequently revert to its original condition. Well supported cloths and woven metals do not show this effect.

The resistance of the filter cloth cannot normally be considered separately from that of the initial layers of cake, because it is difficult to define precisely the boundary between the cloth and the cake. The combined resistance is always considerably greater than the resistance of the clean cloth plus that of the initial layer of cake because the solid particles tend to block the pores of the cloth and to form bridges over the entrances to the channels. With thick slurries, there is a tendency for particles to form bridges over the channel entrances and there is then a smaller tendency for the particles to be carried into the pores. The embedded particles are usually very difficult to remove and their presence may cause the resistance of the cloth to increase by a factor of 20 or 30. Frequently the cloth has to be discarded, not because it is mechanically worn out, but because the resistance has increased to such an extent as to make continued use uneconomic. In some cases the cloth rapidly becomes clogged because particles adhere rigidly to it.

It will be appreciated therefore that it is difficult to treat the flow problem satisfactorily from the theoretical point of view. In practice, it is usual to regard the cloth, plus the initial layers of cake, as equivalent to a thickness of cake. It is important to remember that this initial resistance is dependent on the manner in which the filtration is commenced, as already indicated.

**Flow of Filtrate through the Cloth and Cake Combined**

Suppose the filter cloth and the initial layers of cake to be equivalent to a thickness $L$ of cake as deposited at a later stage in the process. Then if $\Delta P$ is the pressure drop across the cake and cloth combined,

$$\frac{1}{A}\cdot\frac{\mathrm{d}V}{\mathrm{d}t}=\frac{\Delta P}{r\mu(l+L)} \qquad \ldots.(12.11)$$

(compare equation 12.2)

i.e.
$$\frac{\mathrm{d}V}{\mathrm{d}t}=\frac{A\Delta P}{r\mu\left(\dfrac{Vv}{A}+L\right)}=\frac{A^2\Delta P}{vr\mu\left(V+\dfrac{LA}{v}\right)} \qquad \ldots.(12.12)$$

This equation can be integrated between the limits $t = 0$, $V = 0$ and $t = t_1$, $V = V_1$ for constant rate filtration, and $t = t_1$, $V = V_1$ and $t = t$, $V = V$ for a subsequent constant pressure filtration.

For the period of constant rate filtration,

$$\frac{V_1}{t_1} = \frac{A^2\Delta P}{vr\mu\left(V_1 + \dfrac{LA}{v}\right)}$$

i.e.
$$\frac{t_1}{V_1} = \frac{r\mu v}{A^2\Delta P} V_1 + \frac{r\mu L}{A\Delta P} \qquad \ldots.(12.13a)$$

i.e.
$$V_1^2 + \frac{LA}{v} V_1 = \frac{A^2\Delta P}{r\mu v} t_1 \qquad \ldots.(12.13b)$$

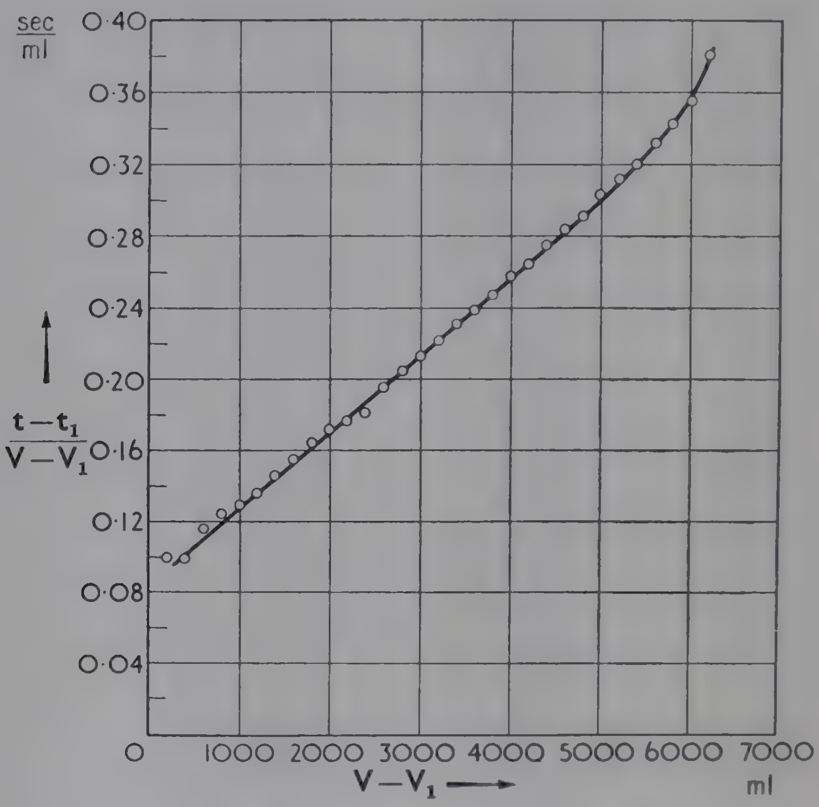


Fig. 12.2. A typical filtration curve

For a constant pressure filtration,

$$\tfrac{1}{2}(V^2 - V_1^2) + \frac{LA}{v}(V - V_1) = \frac{A^2\Delta P}{r\mu v}(t - t_1) \qquad \ldots.(12.14a)$$

i.e.
$$(V - V_1 + 2V_1)(V - V_1) + \frac{2LA}{v}(V - V_1) = \frac{2A^2\Delta P}{r\mu v}(t - t_1)$$

i.e.
$$\frac{t - t_1}{V - V_1} = \frac{r\mu v}{2A^2\Delta P}(V - V_1) + \frac{r\mu v V_1}{A^2\Delta P} + \frac{r\mu L}{A\Delta P} \qquad \ldots.(12.14b)$$

Thus there is a linear relation between $\dfrac{t - t_1}{V - V_1}$ and $V - V_1$ (as shown in Fig. 12.2) and the slope is proportional to the specific resistance, as in the case of the flow of the filtrate through the filter cake alone (cf. equation 12.10*b*), but the line does not go through the origin.

The intercept on the $\frac{t - t_1}{V - V_1}$ axis should enable $L$, the equivalent thickness of the cloth, to be calculated but reproducible results are not obtained because this resistance is critically dependent on the exact manner in which the operation is commenced. The time at which measurement of $V$ and $t$ is commenced does not affect the slope of the curve, only the intercept. It will be noted that a linear relation between $t$ and $V^2$ is no longer obtained when the cloth resistance is appreciable.

### Compressible Filter Cakes

Nearly all filter cakes are compressible to some extent but in many cases the degree of compressibility is so small that the cake can, for practical purposes, be regarded as incompressible. The evidence for compressibility is that the specific resistance is a function of the pressure difference across the cake. Compressibility may be a reversible or an irreversible process. Most filter cakes are inelastic and the greater resistance offered to flow at high pressure differences is caused by the more compact packing of the particles forming the filter cake. Thus the specific resistance of the cake will correspond to that for the highest pressure difference to which the cake is subjected, even though this maximum pressure difference may be maintained for only a short time. It is therefore important that the filtration pressure should not be allowed to exceed the normal operating pressure at any stage. In elastic filter cakes the elasticity is attributable to compression of the particles themselves: they are rarely encountered, but some forms of carbon can give rise to elastic cakes.

Because of the frictional losses arising from the flow of filtrate through the cake, there will be a fluid pressure gradient. The particles will exert a retarding action on the fluid but the fluid will exert a compressing action on the particles and therefore tend to consolidate the cake. Since the force exerted by the fluid on each particle is transmitted to the particles deeper in the cake, the compressive force varies from a minimum of zero at the free surface of the cake to a maximum at the filter medium. The actual compressive pressure will depend on the structure of the cake and the nature of the contacts between the particles, but it can be expressed as a function of the difference between the pressure at the surface of the cake $P_2$ and that at a depth $z$ in the cake $P_z$ (see Fig. 12.3). Thus since the voidage $e_z$ at any point is a function of the compressive force at that point,

$$e_z = f(P_2 - P_z) \qquad \ldots .(12.15)$$

The voidage thus decreases from the free surface to the filter medium and the resistance shows a corresponding increase. If the filtration surface is horizontal and facing upwards, the effect of the fluid drag will be supplemented by gravity. Since the resistance varies throughout the depth of the cake, the filtration equation 12.2 must be expressed in differential form

$$\frac{1}{A}\frac{\mathrm{d}V}{\mathrm{d}t} = \frac{1}{r_z \mu}\frac{\mathrm{d}P_z}{\mathrm{d}z} \qquad \ldots .(12.16)$$

where $r_z$ is the point value of the specific resistance. This equation may be written as

$$\frac{1}{A}\frac{dV}{dt} = \frac{e_z^{\,3}}{5(1-e_z)^2S^2}\,\frac{1}{\mu}\,\frac{dP_z}{dz} \qquad \ldots.(12.17)$$

The small element of cake thickness dz must now be related to the small volume d$V$ of filtrate passing during its formation. As the cake is compressible, the volume of cake deposited by the flow of unit volume of filtrate will not be constant, but the mass of solids deposited $c$ will be almost independent of the conditions under which the cake is formed. We thus have

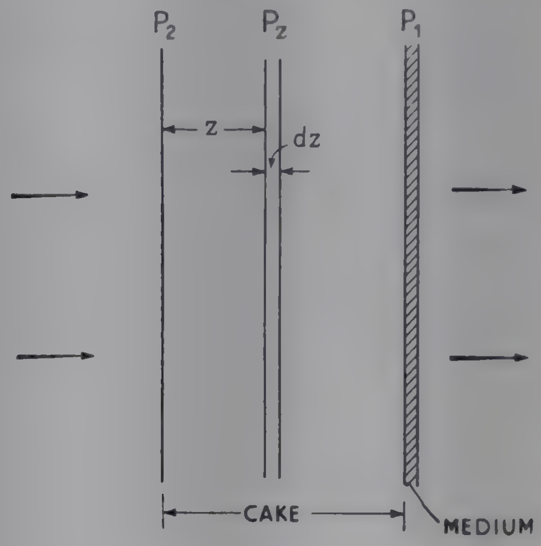


Fig. 12.3. Flow through a compressible filter cake

$$dz = dV\frac{c}{(1-e_z)\rho_s A} \qquad \ldots.(12.18)$$

Thus, from equation 12.17,

$$\frac{1}{A}\frac{dV}{dt} = \frac{e_z^{\,3}}{5(1-e_z)^2S^2}\,\frac{(1-e_z)\rho_s A}{c}\,\frac{1}{\mu}\,\frac{dP_z}{dV}$$

$$\therefore \qquad \frac{dV}{dt} = \frac{e_z^{\,3}\rho_s}{5(1-e_z)S^2}\,\frac{A^2}{\mu c}\,\frac{dP_z}{dV} \qquad \ldots.(12.19)$$

$$= \frac{A^2}{\mu c\bar{r}_z}\,\frac{dP_z}{dV} \qquad \ldots.(12.20)$$

where

$$\bar{r}_z = \frac{5(1-e_z)S^2}{e_z^{\,3}\rho_s} \qquad \ldots.(12.21)$$

The specific resistance term $\bar{r}$ is based on the flow of filtrate through unit mass of cake deposited on unit area, whereas $r$ was based on the flow through a unit cube of cake. Comparison of equations 12.2 and 12.20 shows that for an incompressible cake,

$$c\bar{r} = vr$$

i.e.

$$\bar{r} = r \,.\, \frac{v}{c} \qquad \ldots.(12.22)$$

The dimensions of $\bar{r}$ are therefore $\mathbf{L}^{-2} \times \mathbf{L}^3\mathbf{M}^{-1} = \mathbf{M}^{-1}\mathbf{L}$. For any instant in a constant pressure filtration we have, on integration through the whole depth of the cake,

$$\int_0^V \frac{dV}{dt}\,dV = \frac{A^2}{\mu c}\int_{P_2}^{P_1} \frac{dP_z}{\bar{r}_z}$$

At any instant, $\frac{dV}{dt}$ is constant throughout the cake and therefore

$$\frac{dV}{dt} = \frac{A^2}{V\mu c}\int_{P_2}^{P_1} \frac{dP_z}{\bar{r}_z} \qquad \ldots.(12.23)$$

Now $\bar{r}_z$ has been shown to be a function of the pressure difference $P_2 - P_z$, but is independent of the absolute value of the pressure. Suppose that we can write

$$\bar{r}_z = \bar{r}'\,(P_2 - P_z)^{n'} \qquad \ldots.(12.24)$$

where $\bar{r}'$ is independent of $P_z$

Then,

$$\int_{P_2}^{P_1} \frac{dP_z}{\bar{r}_z} = \frac{1}{\bar{r}'}\int_{P_2}^{P_1} \frac{dP_z}{(P_2 - P_z)^{n'}}$$

$$= \frac{1}{\bar{r}'}\,\frac{(P_2 - P_1)^{1-n'}}{1 - n'} \quad \text{(unless } n' = 1\text{)}$$

$$= \frac{1}{\bar{r}'}\,\frac{\Delta P^{1-n'}}{1 - n'}$$

Thus

$$\frac{dV}{dt} = \frac{A^2}{V\mu c\bar{r}'}\,\frac{\Delta P}{(1 - n')\Delta P^{n'}}$$

$$= \frac{A^2\Delta P}{V\mu c\bar{r}''\Delta P^{n'}} \qquad \ldots.(12.25)$$

(where $\bar{r}'' = (1 - n')\bar{r}'$)

$$= \frac{A^2\Delta P}{V\mu c\bar{r}} \qquad \ldots.(12.26)$$

where $\bar{r}$ is the mean resistance as defined by equation 12.26.

i.e.

$$\bar{r} = \bar{r}''\Delta P^{n'} \qquad \ldots.(12.27)$$

Typical values of $n'$ are 0·01 to 0·15 for precipitated calcium carbonate: this forms a cake of low compressibility. For certain highly compressible hydroxide sludges $n'$ may be as high as 0·9: for these materials, very little increase is obtained in the rate of filtration as a result of increasing the pressure difference. An equation of the form of 12.27 was suggested originally by SPERRY(19), but it is applicable only over a small range of conditions because it gives the resistance as zero when the pressure difference is zero. A more generally applicable expression is due to RUTH(26, 27),

$$\bar{r} = \bar{r}'''(1 + I\Delta P^{n'}) \qquad \ldots.(12.28)$$

For a constant pressure filtration, it is found that equation 12.26 can be directly integrated in the same way as for an incompressible cake, and that the equations for incompressible cakes are valid if $c\bar{r}$ is substituted for $vr$.

GRACE(65) has correlated the degree of compressibility of a filter cake with the flocculation of the particles in the original slurry. Thus, with a highly flocculated slurry, the cake has a comparatively loose structure and low resistance, but the compressibility is high because the flocs readily break down if a compressive force is applied. The degree of flocculation also affects the effective specific surface and therefore the resistance is also affected in this second manner.

CARMAN(32) has shown that there may be a fourfold variation in the resistance of a filter cake throughout its depth. RIETEMA(67) has found that, when the filtering surface is vertical, the bottom of the cake is compressed as a result of gravitational force. This occurs when the cake thickness exceeds a critical value and is shown by a sudden change in the slope of the filtration curve.

The velocity of flow of filtrate may influence the specific resistance of a compressible filter cake, because the orientation of the particles and their compactness will be affected by the velocity with which they are deposited. Further, the higher the velocity the greater is the tendency for the smaller particles to be drawn forward in the cake. Both of these effects will cause the resistance of the cake to increase towards the cloth. DONALD and HUNNEMAN(12) have suggested that the resistance at any point in the cake will be a simple power function of the velocity with which the solids have been deposited. The effect of the velocity of deposition is thus seen to augment the effect of the compressive forces arising from the fluid pressure gradient. According to DONALD and HUNNEMAN, we can write,

$$\bar{r}_z = r''\left(\frac{1}{A}\frac{\mathrm{d}V}{\mathrm{d}t}\right)^n \qquad \ldots .(12.29)$$

Then taking into account the effect of pressure difference on resistance, as indicated by equation 12.28,

$$\bar{r}_z = r_0(1 + I\Delta P^{n'})\left(\frac{1}{A}\frac{\mathrm{d}V}{\mathrm{d}t}\right)^n \qquad \ldots .(12.30)$$

For a constant pressure filtration, we can use equation 12.29, if we remember that $r''$ is a function of $\Delta P$. Since the velocity of deposition of the filter cake varies throughout, equation 12.29 gives only the point effect of the filtrate velocity. The mean value of the resistance is then given by

$$\frac{1}{V}\int_0^V r''\left(\frac{1}{A}\frac{\mathrm{d}V}{\mathrm{d}t}\right)^n \mathrm{d}V$$

Then substituting in equation 12.26,

$$\frac{\mathrm{d}V}{\mathrm{d}t} = \frac{A^2\Delta P}{V\mu c\dfrac{1}{V}\displaystyle\int_0^V r''\left(\frac{1}{A}\frac{\mathrm{d}V}{\mathrm{d}t}\right)^n \mathrm{d}V}$$

$$= \frac{A^{2+n}\Delta P}{\mu c r''\displaystyle\int_0\left(\frac{\mathrm{d}V}{\mathrm{d}t}\right)^n \mathrm{d}V}$$

$$\therefore \qquad \int_0^V\left(\frac{\mathrm{d}V}{\mathrm{d}t}\right)^n \mathrm{d}V = \frac{A^{2+n}\Delta P}{\mu c r''}\frac{\mathrm{d}t}{\mathrm{d}V}$$

Differentiating with respect to $V$,

$$\left(\frac{\mathrm{d}V}{\mathrm{d}t}\right)^n = \frac{A^{2+n}\Delta P}{\mu c r''}\frac{\mathrm{d}^2 t}{\mathrm{d}V^2}$$

$$\therefore \qquad \left(\frac{\mathrm{d}t}{\mathrm{d}V}\right)^{-n} = \frac{A^{2+n}\Delta P}{\mu c r''}\frac{\mathrm{d}}{\mathrm{d}V}\left(\frac{\mathrm{d}t}{\mathrm{d}V}\right)$$

$$\therefore \qquad \int_0^V \mathrm{d}V = \frac{A^{2+n}\Delta P}{\mu c r''}\int_0^{\frac{\mathrm{d}t}{\mathrm{d}V}}\left(\frac{\mathrm{d}t}{\mathrm{d}V}\right)^n \mathrm{d}\left(\frac{\mathrm{d}t}{\mathrm{d}V}\right)$$

$$\therefore \qquad V = \frac{\dfrac{A^{2+n}\Delta P}{\mu c r''}\left(\dfrac{\mathrm{d}t}{\mathrm{d}V}\right)^{n+1}}{n+1}$$

$$\therefore \qquad \mathrm{d}t = \left\{\frac{(n+1)\,\mu c r''}{A^{2+n}\Delta P}V\right\}^{\frac{1}{n+1}}\mathrm{d}V$$

and

$$t = \left\{\frac{(n+1)\,\mu c r''}{A^{2+n}\Delta P}\right\}^{\frac{1}{n+1}} V^{\frac{n+2}{n+1}}\,\frac{n+1}{n+2}$$

i.e.

$$\left\{\frac{n+2}{n+1}t\right\}^{n+1} = \frac{(n+1)\mu c r''}{A^{2+n}\Delta P}V^{n+2} \qquad \ldots.(12.31)$$

i.e.

$$t^{n+1} \propto V^{n+2} \qquad \ldots.(12.32)$$

It is found experimentally that $n$ varies from 0 to about 20. When $n = 0$, i.e. when the velocity has no effect,

$$t \propto V^2 \qquad \ldots.(12.33)$$

as for the incompressible cake.

***Flow through a Compressible Cake and Filter Cloth.*** At first sight one might not expect the resistance of the cloth to be affected by the difference in pressure but, as already explained, particles become embedded in the cloth, which then shows some of the compressibility effects of the cake. Equation 12.28 will therefore apply approximately for the filter cloth but the index $n$ will be less than that for the cake. The problem may be simplified by considering the compressibility of the cloth to be the same as that of the cake, so that the resistance of the cloth is equivalent to that of a certain thickness of cake; alternatively, the cloth resistance can be taken as constant. The latter is the more general procedure and is justifiable in the case of thick felts and flannels. In that case the proportion of the total pressure drop which occurs across the cloth is continuously diminishing and therefore the pressure at the boundary between the cloth and the cake will progressively change during a constant pressure filtration. Thus the limits of equation 12.23 will vary continuously and the equation must therefore be integrated numerically. If the flow through the cloth is given by

$$\frac{1}{A}\frac{\mathrm{d}V}{\mathrm{d}t}=\frac{\Delta P''}{\mu U} \qquad \ldots.(12.34)$$

$$\Delta P''=\frac{\mu U}{A}\frac{\mathrm{d}V}{\mathrm{d}t} \qquad \ldots.(12.35)$$

where $\Delta P''$ is the pressure drop across the cloth, and
$U$ is the resistance of the cloth.

The corresponding limit of the integral will therefore be $P_1+\frac{\mu U}{A}\frac{\mathrm{d}V}{\mathrm{d}t}$.

**Preliminary Treatment of Slurries before Filtration**

If the slurry is dilute and the solid particles settle readily in the fluid, it may be desirable to effect a preliminary concentration in a thickener (see Chapter 15). The thickened suspension is then fed from the thickener to the filter and the quantity of material to be handled is thereby reduced.

The theoretical treatment has shown that the nature of the filter cake has a very pronounced effect on the rate of flow of filtrate and that it is, in general, desirable that the particles forming the filter cake should have as large a size as possible. More rapid filtration is therefore obtained if a suitable agent is added to the slurry to cause coagulation. If the solid material is formed in a chemical reaction by precipitation, the particle size can generally be controlled to a certain extent by the actual conditions of formation. For example, the particle size of the resultant precipitate can be controlled by varying the temperature and concentration, and sometimes the $p_H$, of the reacting solutions. As indicated by GRACE, a flocculated suspension gives rise to a more porous cake but the compressibility is greater. In many cases, crystal shape can be altered by adding traces of material which is selectively adsorbed on particular faces (see Chapter 23).

Filter aids are extensively used where the filter cake is relatively impermeable to the flow of filtrate. They are materials which pack to form beds of very high voidages and therefore they are capable of increasing the porosity of the filter cake if added to the slurry before filtration. Apart from economic considerations, there is an optimum quantity of filter aid which should be added in any given case. Whereas the presence of the filter aid reduces the specific resistance of the filter cake, it also results in the formation of a thicker cake. The actual quantity used will therefore depend on the nature of the material. The use of filter aids is normally restricted to operations in which the filtrate is valuable and the residue is a waste product. In some circumstances, however, the filter aid must be readily separable from the rest of the filter cake by physical or chemical means. Filter cakes incorporating filter aid are usually very compressible and care should therefore be taken to ensure that the good effect of the filter aid is not destroyed by employing too high a filtration pressure. Kieselguhr, which is a commonly used filter aid, has a voidage of about 0·85. Addition of relatively small quantities increases the voidage of most filter cakes; the resulting porosity normally lies between that of the filter aid and that of the filter solids. Sometimes the filter medium is "precoated" with filter aid, and a thin layer of the filter aid is removed with the cake at the end of each cycle.

In some cases the filtration time can be reduced by diluting the suspension in order to reduce the viscosity of the filtrate. This does, of course, increase the bulk to be filtered and is applicable only when the value of the filtrate is not affected by dilution.

### Washing of the Filter Cake

When the wash liquid is miscible with the filtrate and has similar physical properties, the rate of washing at the same pressure difference will be about the same as the final rate of filtration. If the viscosity of the wash liquid is less, a somewhat greater rate will be obtained. However, channelling sometimes occurs, with the result that much of the cake is incompletely washed; the fluid passing preferentially through the channels, which are gradually enlarged by its continued passage. This does not occur during filtration because channels are self-sealing by virtue of deposition of solids from the slurry. Channelling is most marked with compressible filter cakes and can be minimised by using a smaller pressure difference for washing than for filtration.

Washing can be regarded as taking place in two stages. First, filtrate is displaced from the filter cake by wash liquid during the period of "displacement washing"; in this way up to 90% of the filtrate may be removed. During the second stage, "diffusional washing," solvent diffuses into the wash liquid from the less accessible voids and the following relation applies

$$\frac{\text{volume of wash liquid passed}}{\text{cake thickness}} = \text{a constant} \times \log \frac{\text{initial concentration of solute}}{\text{concentration at particular time}} \quad \ldots.(12.36)$$

Although an immiscible liquid is seldom used for washing, air is often used to effect partial drying of the filter cake. The rate of flow of air must normally be determined experimentally, though BROWNELL(44) does employ the method quoted on page 404.

## FILTRATION EQUIPMENT

The most suitable filter for any given operation is the one which will fulfil the requirements at the minimum overall cost. Since the cost of the equipment will be closely related to the filtering area, it is normally desirable to obtain a high overall rate of filtration. This involves the use of relatively high pressures but the maximum pressures are often limited by considerations of mechanical design. Although a higher throughput from a given filtering surface is obtained from a continuous filter than from a batch operated filter, it may sometimes be necessary to use a batch filter, particularly if the filter cake has a high resistance, because most continuous filters operate under reduced pressure and the maximum filtration pressure is therefore limited. Other features which are desirable in a filter include ease of discharge of the filter cake in a convenient physical form, and a method of observing the quality of the filtrate obtained from each section of the plant. These factors will be kept to the fore in the discussion of the types of equipment available. The most important types which will be referred to are filter presses, leaf filters, and continuous rotary filters. In addition, mention will be made of filters for special purposes, such as bag filters, and the disc type of filter which is used for the removal of small quantities of solids from a fluid.

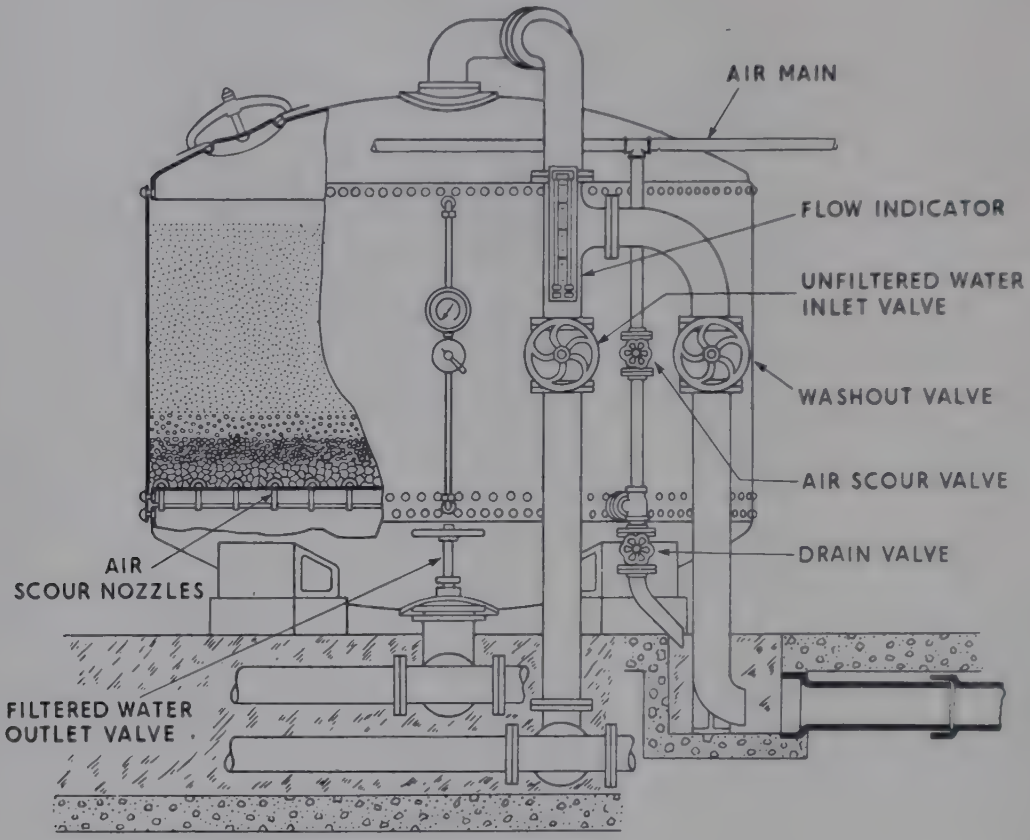


Fig. 12.4. A bed filter

## Bed Filters

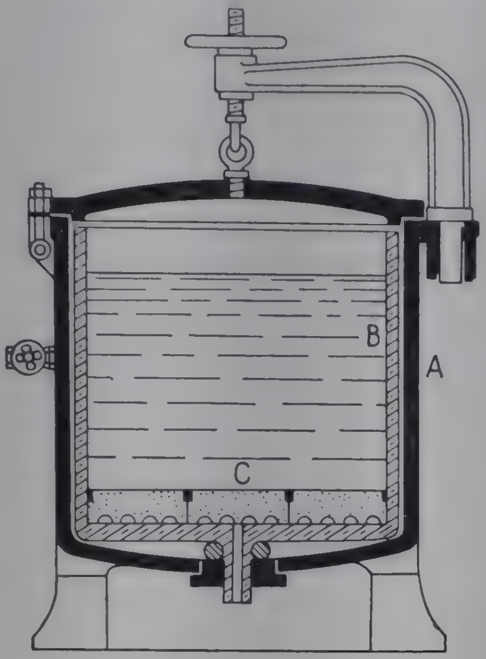
A bed filter operating under positive pressure is illustrated in Fig. 12.4 from which it is seen that the filter medium consists of a number of layers of closely graded solids, usually sand and gravel, supported on a perforated metal screen or on slats. The liquid is fed in at the top and a baffle prevents erosion of the top layers of the bed. Filtration is continued until the resistance has built up to such a level that the rate of filtration is too slow. The bed is then cleaned by scouring with compressed air and an upward current of liquid carries the solids away. Alternatively, the top layers of the bed may be replaced with fresh material. In some cases, bed filters operate with a gravity feed.

This type of filter is extensively used for filtration of water supplies and the operation is then improved by the addition of a small quantity of ferrous or aluminium sulphate to the water. This gives a precipitate of hydroxide which forms a flocculant layer over the surface of the bed so that very small particles are retained. Caustic soda solutions are often filtered through a bed of coke or coal and sugar solutions through charcoal which at the same time removes any colouring matter.

The wood wool or asbestos pulp filter is commonly used for the filtration of boiler feed-waters where the amount of solid to be removed is very small. It consists of a large baffled vessel the compartments of which are packed with wood wool through which the liquid flows under gravity. Operation is continued until the rate of filtration falls off; the wood wool is then replaced.

### Porous Solid Filters

Rigid porous filter media can be constructed in a wide range of shapes and sizes and are usually made from brick, sand, alumina or porous carbon. The addition of a small amount of flour before firing ensures the formation of a porous structure. The medium is extremely fragile and must be carefully supported inside a suitable vessel (Fig. 12.5) and accurate grinding of the supporting surfaces is essential; filtration pressures above 60 lb/sq. in. cannot be used. The great disadvantage of this type of filter is that the porous solid soon clogs and is very difficult to clean. Its main use is for strong acid sludges, which are too corrosive for treatment in other types of filters, and in the production of sterile air.



*A* FILTER; B, FUNNEL; C, PLATES

Fig. 12.5. A porous solid filter

### Bag Filters

Bag filters have now been almost entirely superseded for liquid filtration by other types of filter, but one of the few remaining types is the Taylor bag filter (Fig. 12.6) which has been widely used in the sugar industry. A number of long thin bags are attached to a horizontal feed tray and the liquid flows under the action of gravity so that the rate of filtration per unit area is very low; it is possible, however, to arrange a large filtering area in the plant, e.g. up to about 7,500 square feet. The filter is usually arranged in two sections so that each can be inspected separately without interrupting the operation.

Bag filters are still extensively used for the removal of dust particles from gases and can be operated either as pressure filters or as suction filters. In the former case the fan precedes the filter and therefore is subject to the wearing action of any abrasive dust that is present in the gas. The suction filter obviates this difficulty but offers a smaller pressure difference, and must be used if the dust particles form an explosive mixture with the gas as the fan motor is always a potential source of ignition.

The rate of filtration will depend on the material of the bags and the amount and properties of the solids to be removed. In general, the maintenance of an adequate rate of flow is conditional upon the operation of a satisfactory process for freeing the bags from dust at suitable intervals. A large number of devices have been developed for this and the simplest consists of a periodical reversal of the direction of flow of gas through the filter medium. This method has the disadvantage, however, that filtration is interrupted while the bags are cleaned.

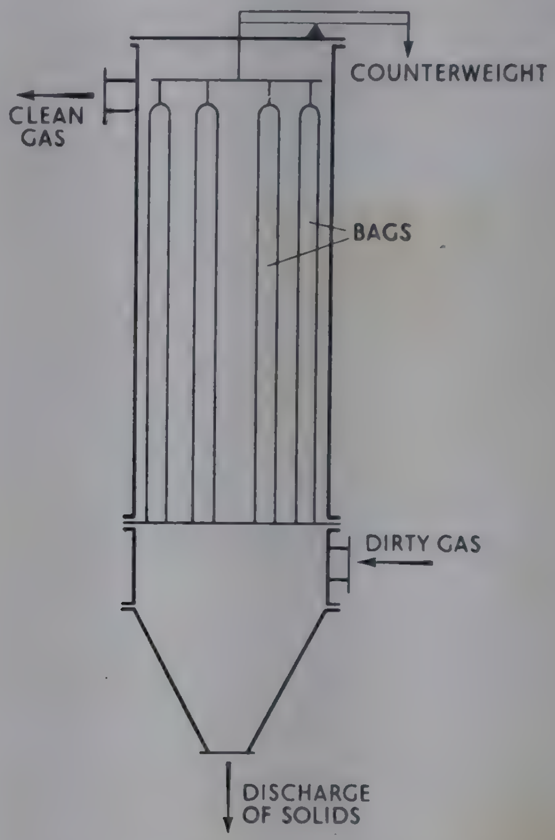


Fig. 12.7. Bag filter with automatic cleaning

A typical dust filter incorporating a mechanism for the automatic cleaning of the bags is shown in Fig. 12.7 and consists of a vertical cylindrical vessel fitted with a horizontal partition to which the bags are attached. A counterweight is so adjusted that the bags are initially under slight tension. As dust collects, the weight of the bags increases and they eventually collapse. The dust is then discharged and the bags become lighter so that they are then brought back under tension again. The solid particles are removed at intervals from the bottom of the hopper. Various other forms of bag cleaner are used and many of them are arranged so that they shake the dust from the bags. The filter illustrated in Fig. 12.8 incorporates a motor-driven shaking device for cleaning the bags.

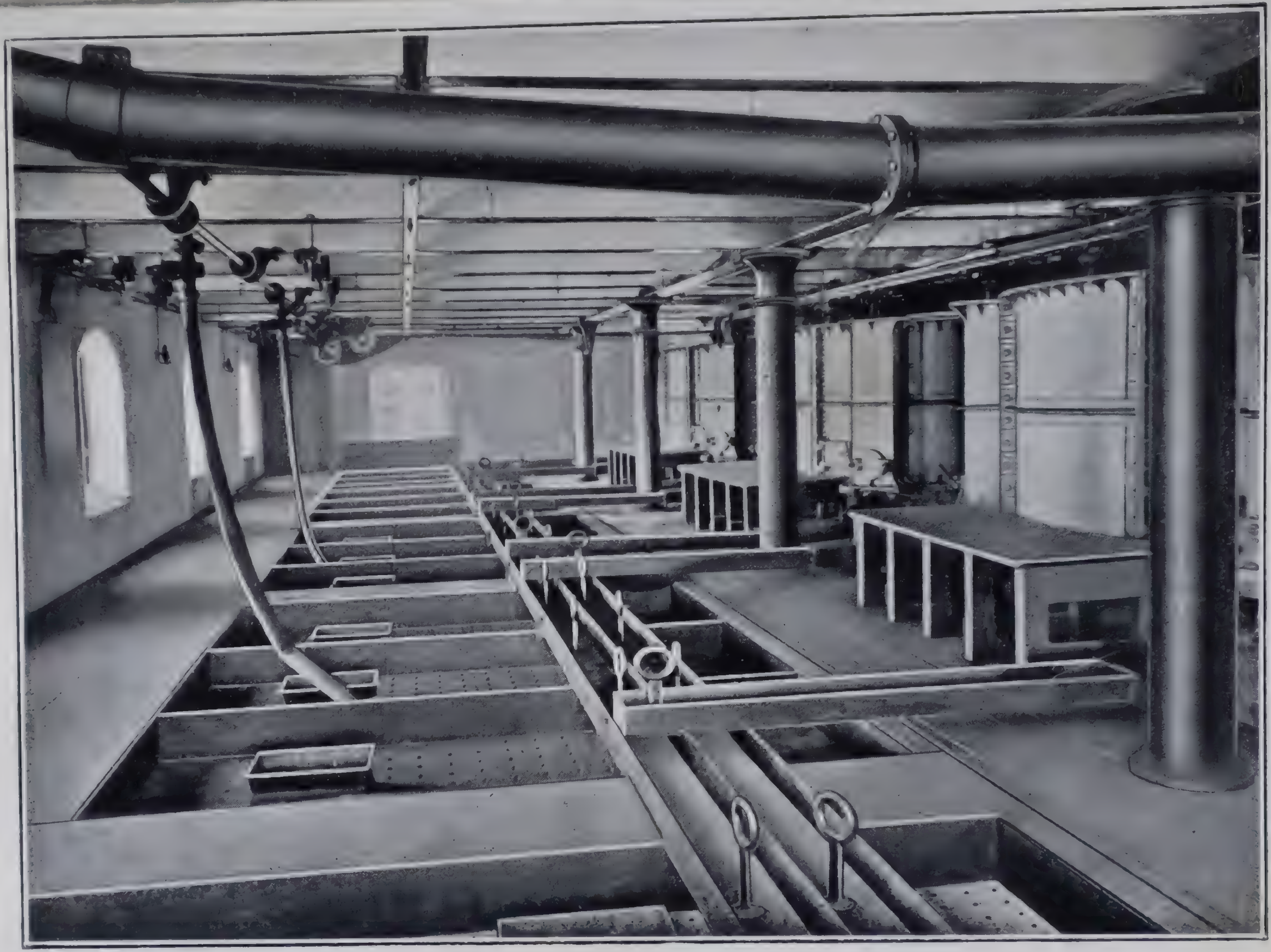


Fig. 12.6. Taylor bag filter at a sugar refinery

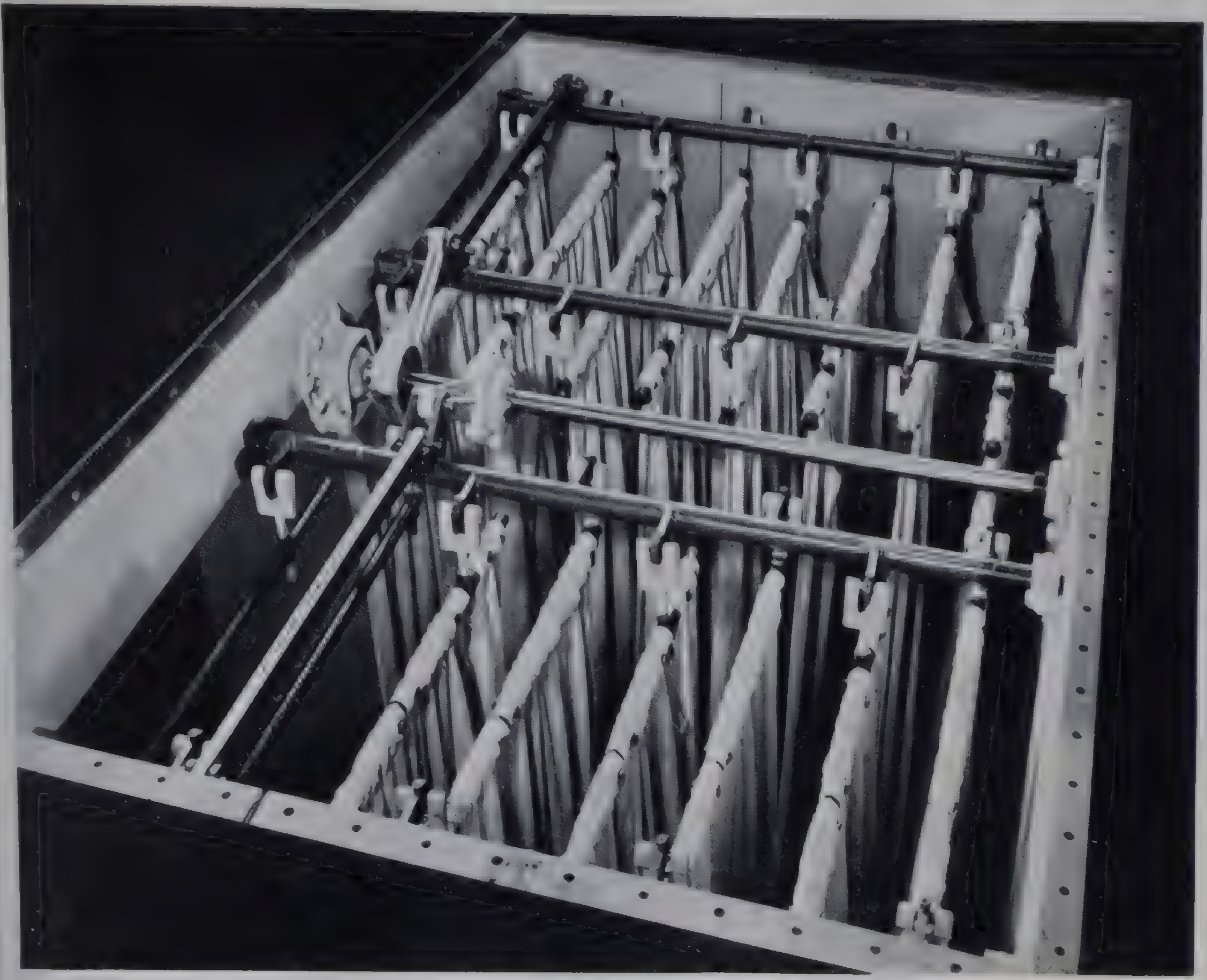


Fig. 12.8. A bag filter unit

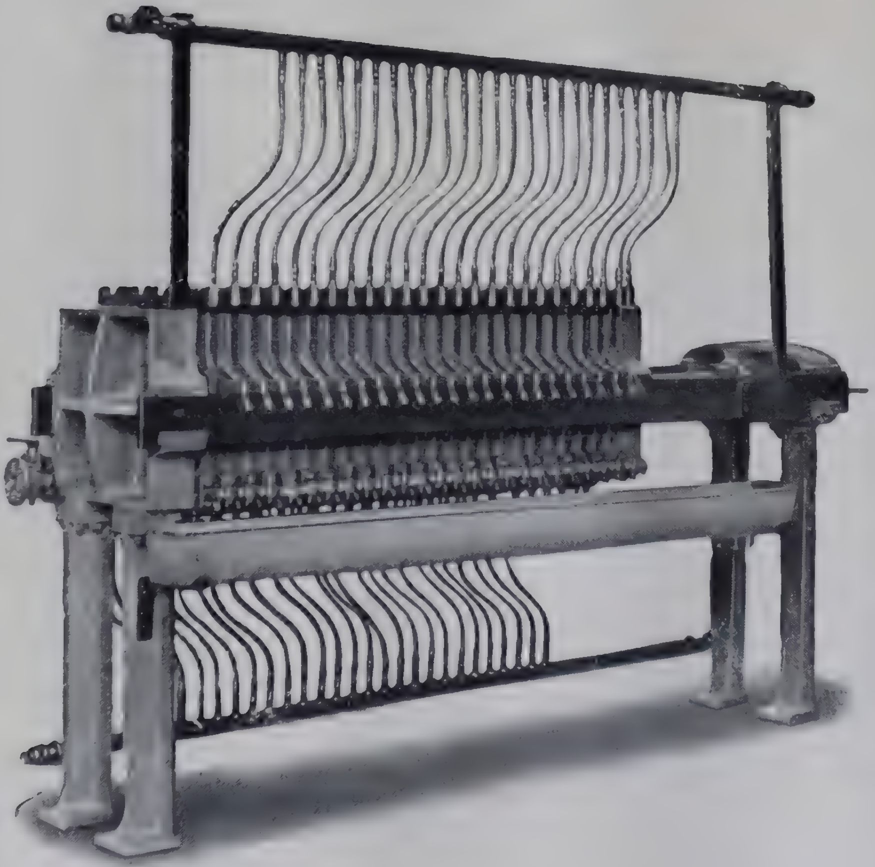


Fig. 12.9. Assembled plate and frame press (steam jacketed)

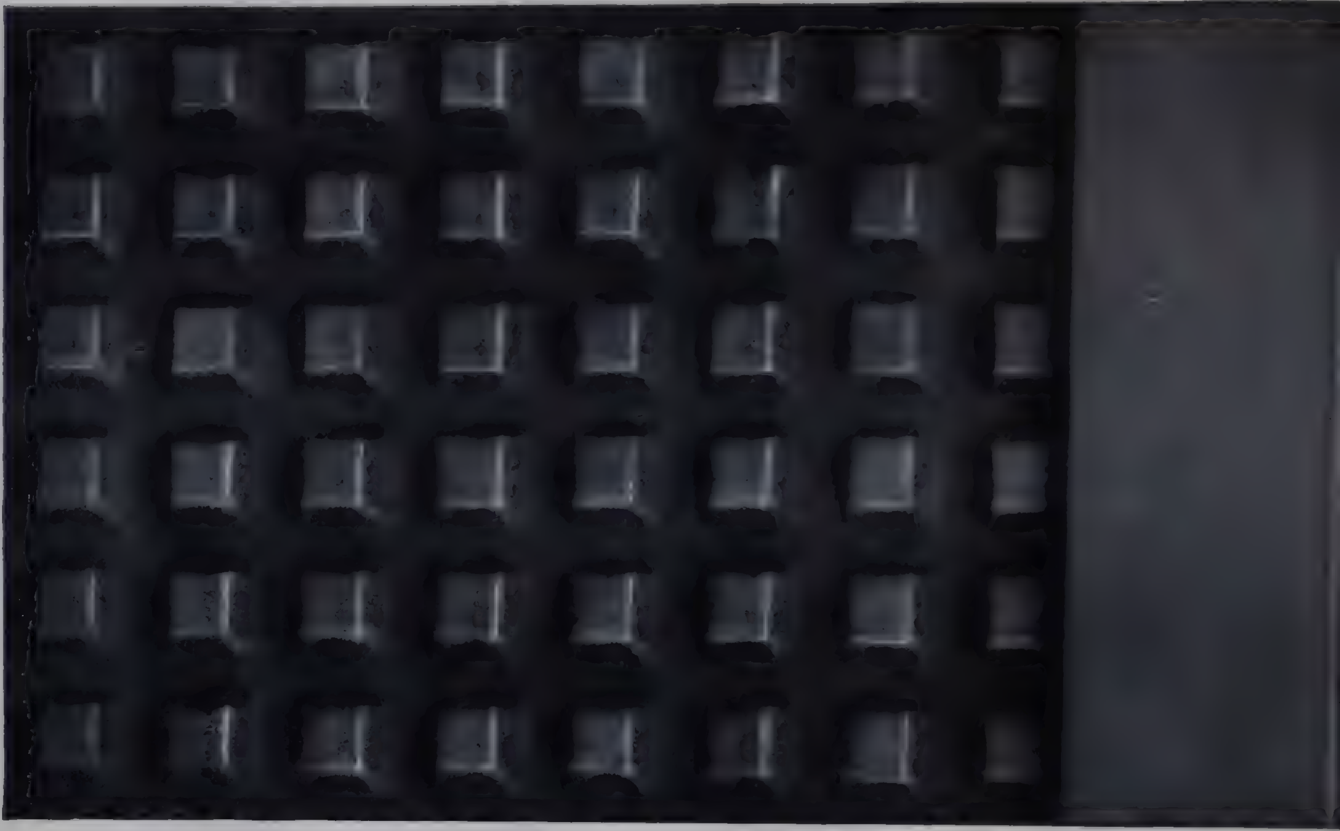


Fig. 12.10. Surface of filter plate

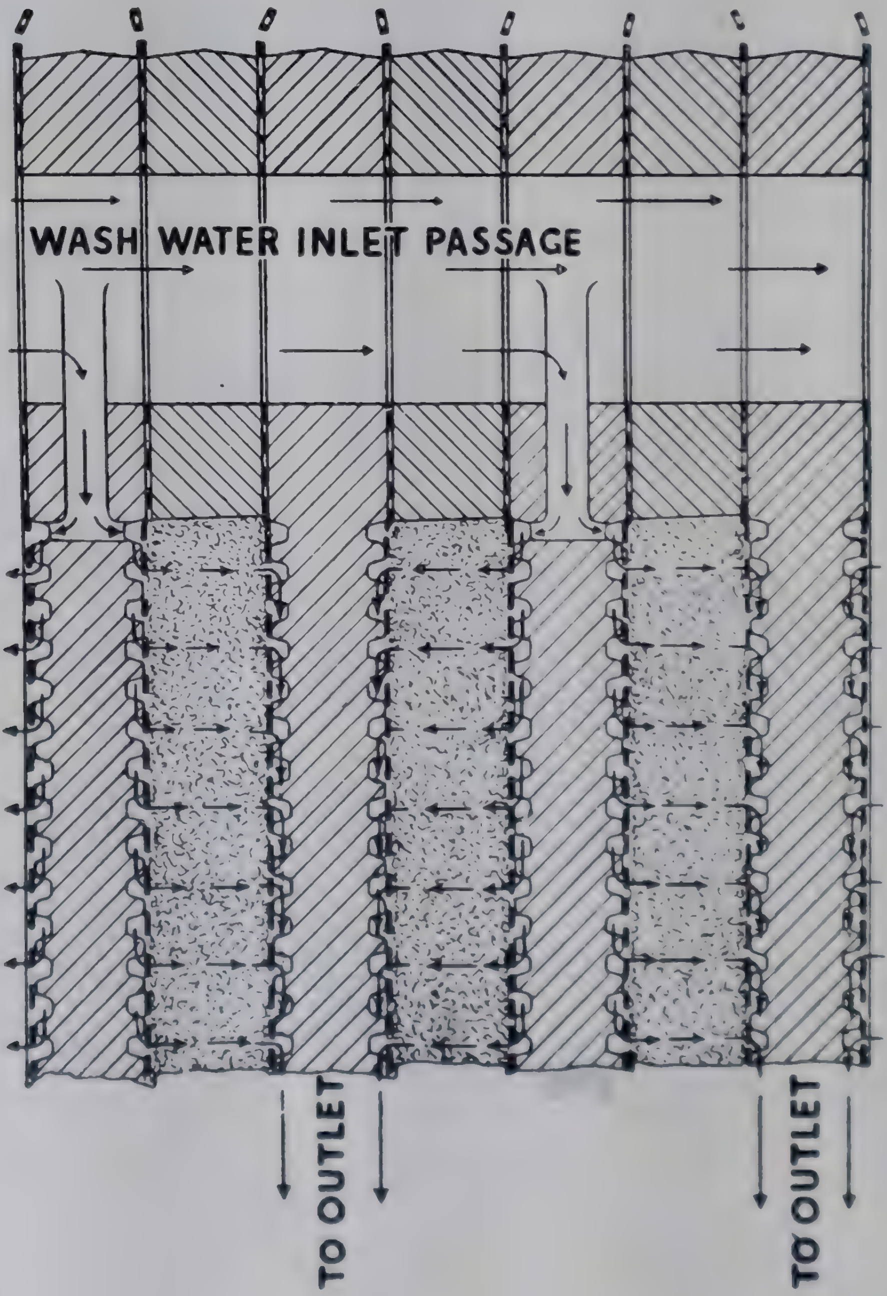

WASH WATER INLET PASSAGE
TO OUTLET
TO OUTLET

Fig. 12.12. Thorough washing

## The Filter Press

The filter press is made in two main forms, the plate and frame press and the recessed plate or chamber press.

***The Plate and Frame Press.*** This type of filter consists of plates and frames arranged alternately and supported on a pair of rails as shown in Fig. 12.9. The plate has a ribbed surface (Fig. 12.10) and the edge stands slightly proud and is carefully machined. The hollow frame is separated from the plate by the filter cloth, and the press is closed either by means of a hand screw or hydraulically; the minimum pressure should be used in order to reduce wear on the cloths. A chamber is therefore formed between each pair of successive plates (Fig. 12.11). The slurry is introduced through a port in each frame and the filtrate passes through the cloth on each side so that two cakes are formed simultaneously in each chamber, and these join when the frame is full. The frames are usually square and may be between 4 in. and 4 ft across and from $\frac{1}{2}$ in. to 3 in. thick.

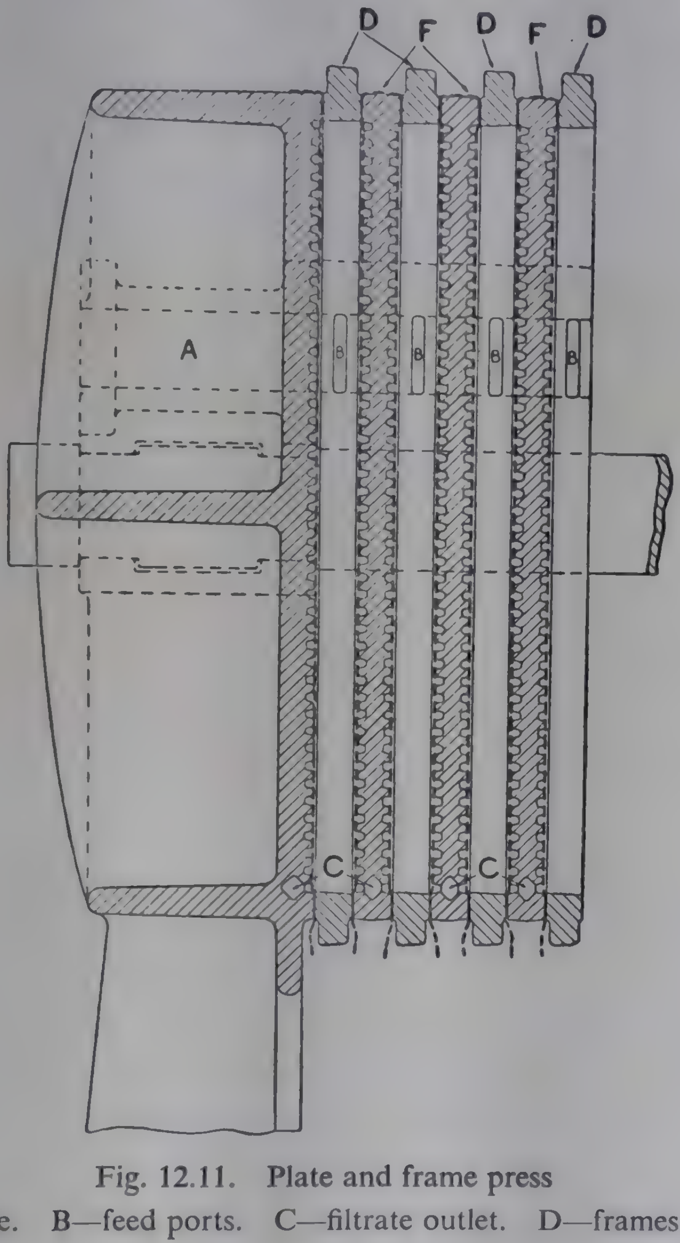


Fig. 12.11. Plate and frame press
A—inlet passage. B—feed ports. C—filtrate outlet. D—frames. F—plates

The slurry may be fed to the press through the continuous channel formed by the holes in the corners of the plates and frames, in which case it is necessary to cut corresponding holes in the cloths which themselves act as gaskets. Cutting of the cloth can be avoided by feeding through a channel at the side but rubber bushes must then be fitted so that a leak-tight joint is formed.

The filtrate runs down the ribbed surface of the plates and then is discharged through a cock into an open launder so that the filtrate from each plate can be inspected and any plate can be isolated if it is not giving a clear filtrate. In some cases the filtrate is removed through a closed channel but it is not then possible to observe the discharge from each plate separately.

In many filter presses, provision is made for steam heating (as shown in Fig. 12.9) so that the viscosity of the filtrate is reduced and a higher rate of filtration obtained. Materials, such as waxes, that solidify at normal temperatures, can also be filtered in steam-heated presses. Steam heating also facilitates the production of a dry cake.

*Optimum Time Cycle*

The optimum thickness of cake to be formed in a filter press depends on the resistance offered by the filter cake and on the time taken to dismantle and refit the press. Although the production of a thin filter cake results in a high average rate of filtration, it is necessary to dismantle the press more often and a greater time is therefore spent on this operation. For a filtration carried out entirely at constant pressure

$$\frac{t}{V} = \frac{r\mu v}{2A^2\Delta P} V + \frac{r\mu L}{A\Delta P} \qquad \ldots .(12.37)$$

(from equation 12.14*b*)

$$= B_1 V + B_2 \qquad \ldots .(12.38)$$

(where $B_1$ and $B_2$ are constant)

Thus the time of filtration $t$ is given by,

$$t = B_1 V^2 + B_2 V. \qquad \ldots .(12.39)$$

The time of dismantling and assembling the press ($t'$, say) is substantially independent of the thickness of cake produced. The total time of a cycle in which a volume $V$ of filtrate is collected is then $t + t'$ and the overall rate of filtration is given by,

$$W = \frac{V}{B_1 V^2 + B_2 V + t'}$$

$W$ is a maximum when $\dfrac{dW}{dV} = 0$.

Differentiating $W$ with respect to $V$ and equating to zero, i.e.

$$B_1 V^2 + B_2 V + t' - V(2B_1 V + B_2) = 0.$$

i.e. $$t' = B_1 V^2 \qquad \ldots .(12.40a)$$

i.e. $$V = \sqrt{\frac{t'}{B_1}} \qquad \ldots .(12.40b)$$

If the resistance of the filter medium is neglected, $t = B_1 V^2$ and the time during which filtration is carried out is exactly equal to the time the press is out of service. In practice, in order to obtain the maximum overall rate of filtration, the filtration time must always be somewhat greater in order to allow for the resistance of the cloth (represented by the term $B_2 V$). In general, the lower the specific resistance of the cake, the greater will be the economic thickness of the frame.

### *Washing*

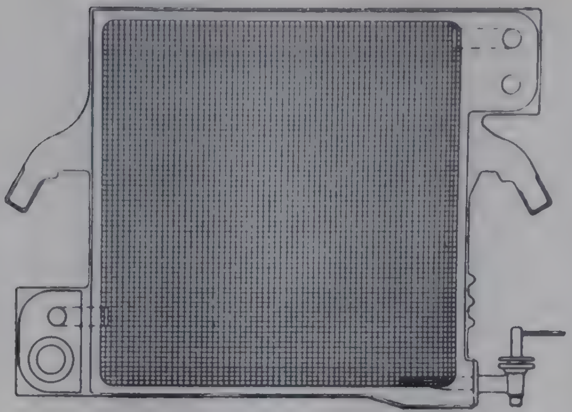
Two methods of washing can be employed, "simple" washing and "through" or "thorough" washing. With simple washing, the wash liquid is fed in through the same channel as the slurry but, as its velocity near the point of entry is high, erosion of the cake takes place. The channels which are thus formed gradually enlarge and uneven washing is usually obtained.

In thorough washing, the wash liquid is introduced through a separate channel behind the filter cloth on alternate plates, known as washing plates (Fig. 12.12), and flows through the whole thickness of the cake, first in the opposite direction and then in the same direction as the filtrate. The area during washing is one-half of that during filtration and, in addition, the wash liquid has to flow through twice the thickness, so that the rate of washing should therefore be about one-quarter of the final rate of filtration. The wash liquid is usually discharged through the same channel as the filtrate though sometimes a separate outlet is provided. Even with thorough washing some channelling occurs and several inlets are often provided so that the liquid is well distributed. If the cake is appreciably compressible, the minimum pressure should be used during washing, and in no case should the final filtration pressure be exceeded. After washing, the cake may be made easier to handle by removing excess liquid with compressed air.

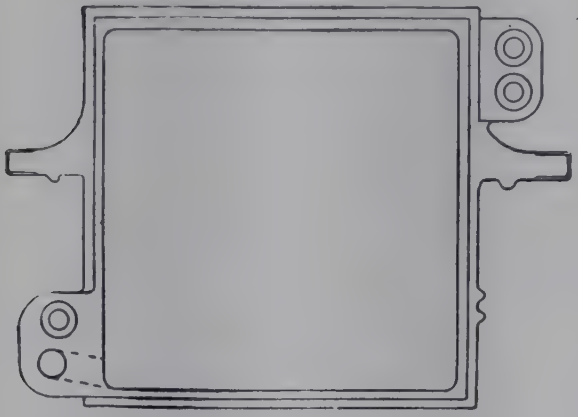
For ease in identification, small buttons are embossed on the sides of the plates and frames, one on the non-washing plates, two on the frames and three on the washing plates. (Fig. 12.13.)

***The Chamber Press.*** The chamber press (Fig. 12.14) is similar to the plate and frame type except that the use of frames is obviated by recessing the ribbed surface of the plates so that the individual filter chambers are formed between successive plates. In this type of press therefore the thickness of the cake cannot be varied.

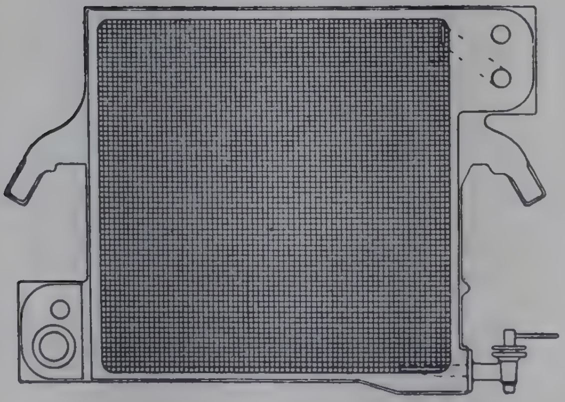
The feed channel (Fig. 12.15) usually differs from that employed on the plate and frame press. All the chambers are connected by means of a comparatively large hole in the centre of each of the plates and the cloths are secured in position by means of screwed unions. Slurries containing relatively large solid particles can readily be handled in this type of press without fear of blocking the feed channels.



One button plate



Distance frame



Three button plate

Fig. 12.13. Plates and frames

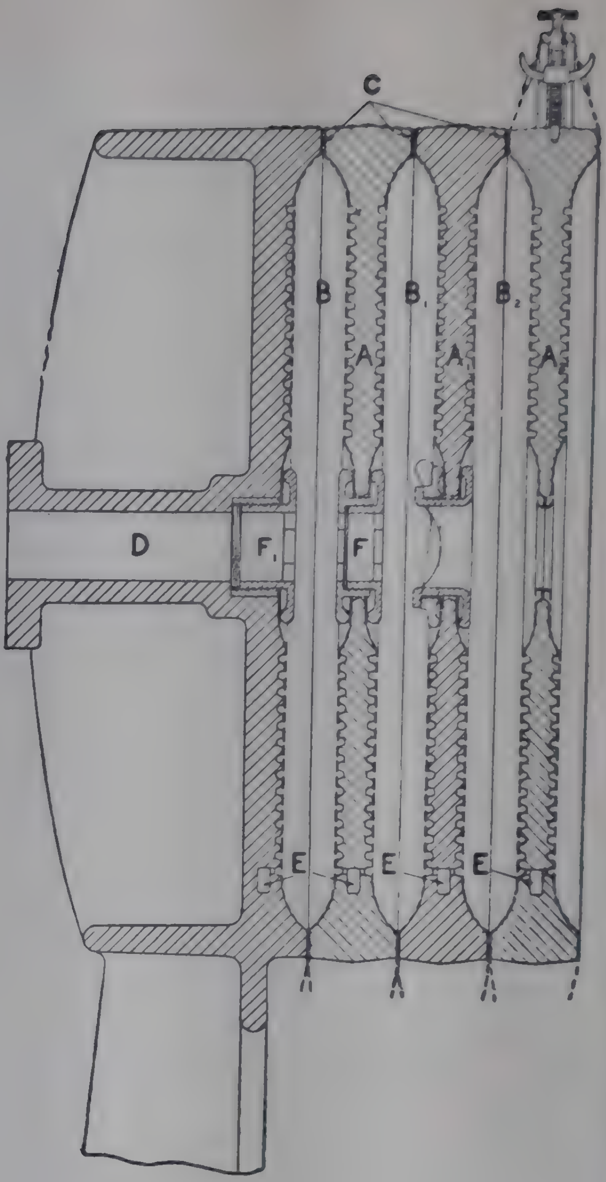


Fig. 12.14. Chamber press

A—plates B—chambers

C—machined rims of plates

D—inlet E—outlets

F—screwed unions

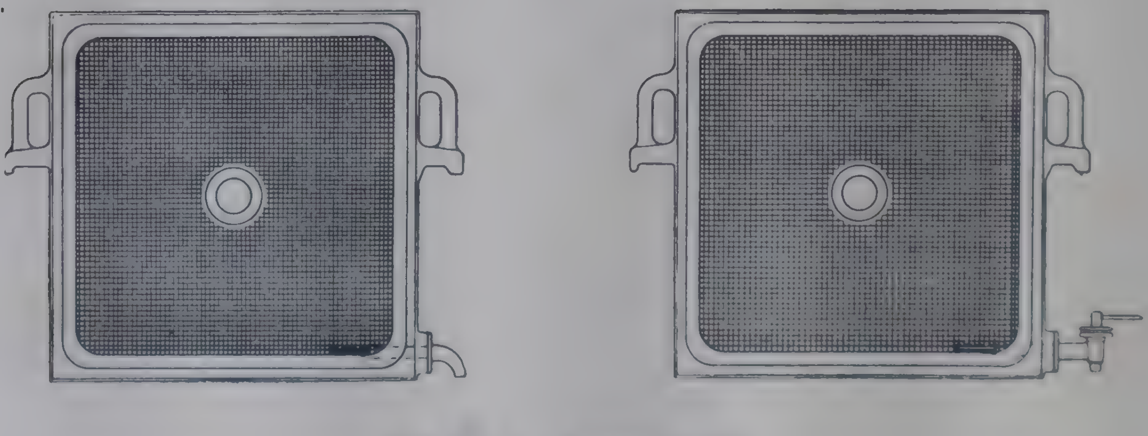


Fig. 12.15. Recessed plate

The chamber press is cheaper in construction than the plate and frame press but clothing is more difficult and greater wear and tear on the cloths results from the distortion produced in all but the shallowest of plates. It is not usual to equip the press with a channel enabling "thorough" washing to be carried out; it is therefore normally used in cases where the solid is of little value and does not require washing. Owing to the greater difficulty in clothing the press, the equipment is usually employed for slurries with a relatively small solid content, i.e. in cases where it is not necessary to dismantle the press frequently.

*Materials of Construction.* Cast iron is the most common material of construction for filter presses, but the castings need careful machining. For small presses which are used only intermittently gun metal and bronze are fairly extensively used. Silver plated materials and stainless steels are used in presses for the pharmaceutical industry and in cases where very slight contamination of the material is likely to be deleterious. Solutions containing hydrochloric acid are frequently filtered in ebonite or enamelled cast iron equipment but this is quite easily chipped. Presses are also constructed of cast iron coated with lead, aluminium alloys, nickel, tin, zinc and some plastic materials.

Wooden presses are in use in the food industries and are commonly employed for filtering cider: they are occasionally used for filtering acid solutions. Their construction is often quite complicated, as many as seven or eight different woods being used in the construction of one press. They should be kept wet in order to prevent shrinkage unless the wood is impregnated with wax. Oak presses are often used for the filtration of acetic acid.

*Advantages of the Filter Press.*

(1) It is simple in construction and can be made from a wide range of materials.

(2) It has no moving parts and in consequence the costs of maintenance and power are low.

(3) It provides a large filtering area in a small floor space.

(4) The capacity and the cake thickness, in the plate and frame press, can be varied by altering the number and the size of the frames.

(5) All joints are external and leakage is readily detected.

(6) High filtration pressures (up to 250 lb/sq. in.) can be used.

(7) The filter cloths are readily replaced on plate and frame presses.

*Disadvantages of the Filter Press.*

(1) It is intermittent in operation and, as the result of frequent dismantling, the wear on the cloths is high, particularly in the chamber press.

(2) The chambers must be filled before the cake can be washed or the press emptied, otherwise handling becomes difficult.

(3) Labour costs are high and therefore the filter press is usually used where the value of the solid product is high, or where the proportion of solids is low.

## Leaf Filters

Many filters incorporate a number of individual leaves, each consisting of a metal framework or a grooved plate over which the filter cloth is fixed. A typical leaf is illustrated in Fig. 12.16. The leaves are immersed in the slurry, which is either fed into the filter under pressure or sucked in by means of a vacuum pump. In either case, the solids build up outside and the filtrate passes through the cloth and is withdrawn from the leaf.

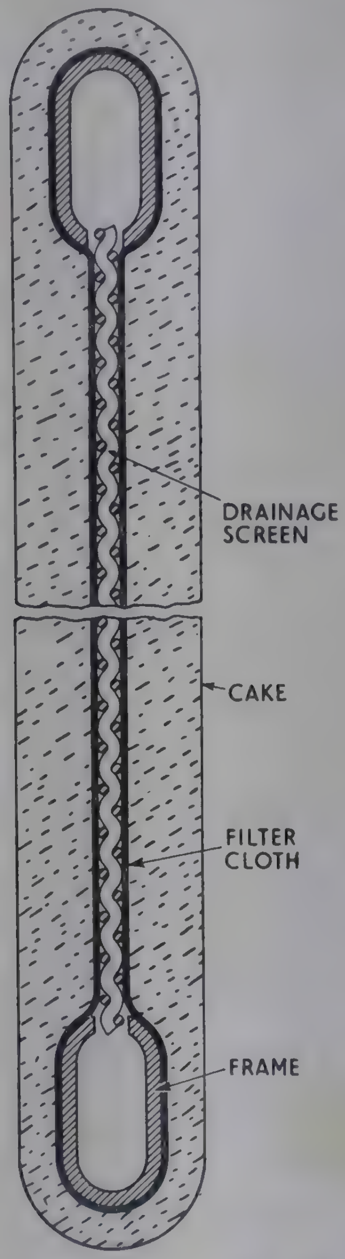


Fig. 12.16. Filter leaf from a Kelly filter

***The Moore Filter.*** The Moore filter is the original form of leaf filter and consists of a number of leaves, sometimes of very large areas, supported on a rigid framework and with the filtrate discharge pipes connected to a common manifold. The battery of leaves is immersed in a tank of slurry and a vacuum is applied through the discharge manifold and filtration is continued until the required thickness of cake has been formed; the thickness is usually between $\frac{1}{8}$ in. and $\frac{3}{8}$ in. The leaf assembly is then lifted out of the slurry tank and immersed in a second tank containing wash liquid. If the filter cake is of little value, a little fine mud may be introduced into the wash liquid to seal any cracks. After washing is complete, the cake is partially dried by sucking air through it, and then loosened from the filter cloth by the application of compressed air internally so that it is readily scraped off. Alternatively, the slurry is withdrawn from the tank at the end of the filtration and is replaced with wash liquid so that it is not necessary to move the leaf assembly.

The filter operates under reduced pressure, so that the pressure difference is limited and hot liquids cannot be filtered because they tend to boil. The Moore filter is now rarely used.

***The Kelly Filter.*** In the Kelly filter, a number of vertical rectangular leaves are arranged longitudinally in a horizontal cylindrical shell. The ends can be opened so that the battery of leaves can be slid out on a pair of rails (Fig. 12.17). The output from each leaf can be observed and controlled independently.

The slurry is pumped into the cylindrical casing and the air is vented to the atmosphere, after which filtration is carried out until a cake of the required thickness is formed; if the slurry tends to sediment, it can be agitated by continuously circulating with a pump. The slurry is then withdrawn and replaced by wash liquid; during this operation, the pressure in the casing is

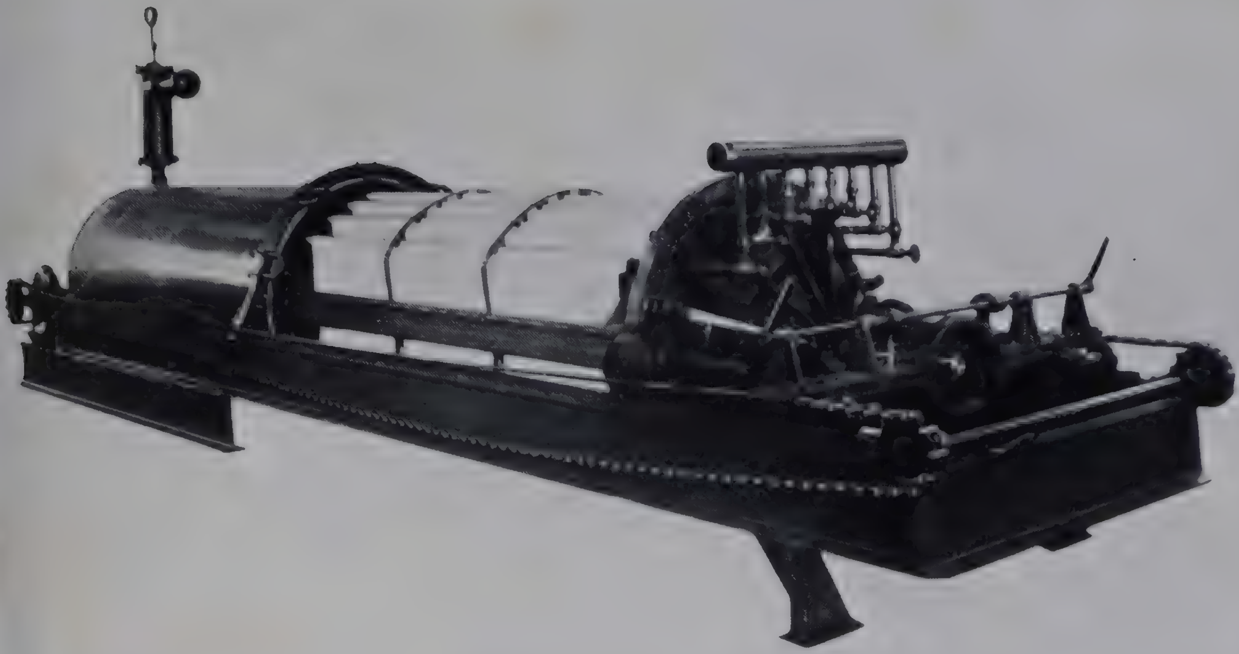


Fig. 12.17. Kelly filter

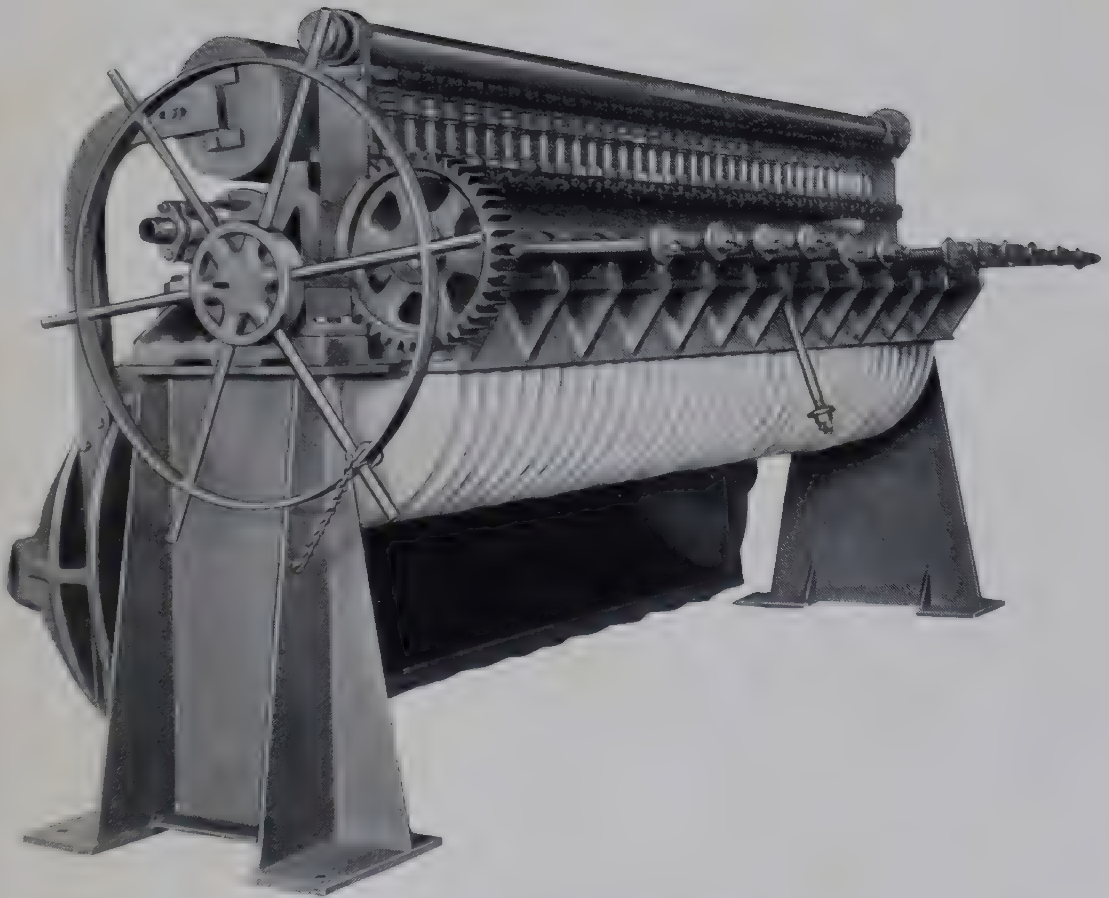


Fig. 12.18. Sweetland filter

maintained with compressed air in order to prevent the cake from cracking. The cake may then be partially dried with air and discharged after the leaves have been withdrawn from the shell; the application of compressed air to the inside of the leaves helps to loosen the cakes.

The Kelly filter contains few leaves and because these are rectangular in shape it is easy to clothe and economical in the use of filter cloth. However, because the leaves are unequal in size, the cakes tend to be uneven; further, the slide rails and rollers may quickly corrode. The filter is usually constructed in mild steel and operates at pressures up to 100 lb/sq. in. Steam heated filters are now used for filtration of liquid sulphur in sulphuric acid plants.

***The Sweetland Filter.*** The Sweetland filter employs circular leaves, all the same size, mounted transversely in a horizontal shell, which is split into two halves with the lower portion hinged so as to give easy access to the leaves (Fig. 12.18). The filtrate from each leaf passes through a sight glass and then enters a common manifold through which the liquid leaves the filter. The operating cycle is similar to that for the Kelly filter, except that the cake is discharged without removing the leaves.

Many Sweetland filters are fitted with a perforated pipe from which the wash water can be sprayed on to the cakes. By this means the cakes are broken up and then reformed by filtering the new slurry that has been produced in the shell. This method of washing is particularly useful in cases where the cake is liable to cracking, which causes the wash water to bypass the bulk of the material. It can also be used to sluice any adhering solids from the cloths.

The Sweetland filter overcomes most of the disadvantages inherent in the other leaf filters, though it is not used at such high pressures as the Kelly filter because it is usually made of cast iron instead of mild steel. Corrosion is a less serious problem because the leaves are readily accessible and the solids can be discharged without moving the leaves. Cakes of any desired thickness can be formed and the filter is therefore very suitable where the cake has a high specific resistance. Very efficient washing of the cakes can be effected. The main drawbacks to the Sweetland filter are its high initial cost and the difficulty of clothing the circular leaves.

***The Vallez Filter.*** This filter is similar to the Sweetland filter in that it employs transverse vertical leaves in a cylindrical casing (Fig. 12.19). The circular leaves (Fig. 12.20) are mounted on a hollow shaft which is slowly rotated at between 1 and 2 r.p.m. The central portion of the leaf is in the form of a hub which is wide enough to give the correct leaf spacing. Ports in the hubs are so placed that they correspond with similar ports on the shaft, which also functions as the filtrate outlet.

The slurry is introduced into the shell of the filter and filtration is continued until the correct thickness of cake has been formed. This is determined by the use of a mechanical gauge. The cake is then washed and air dried, as in the other forms of leaf filter, and the leaves are kept rotating during these operations.

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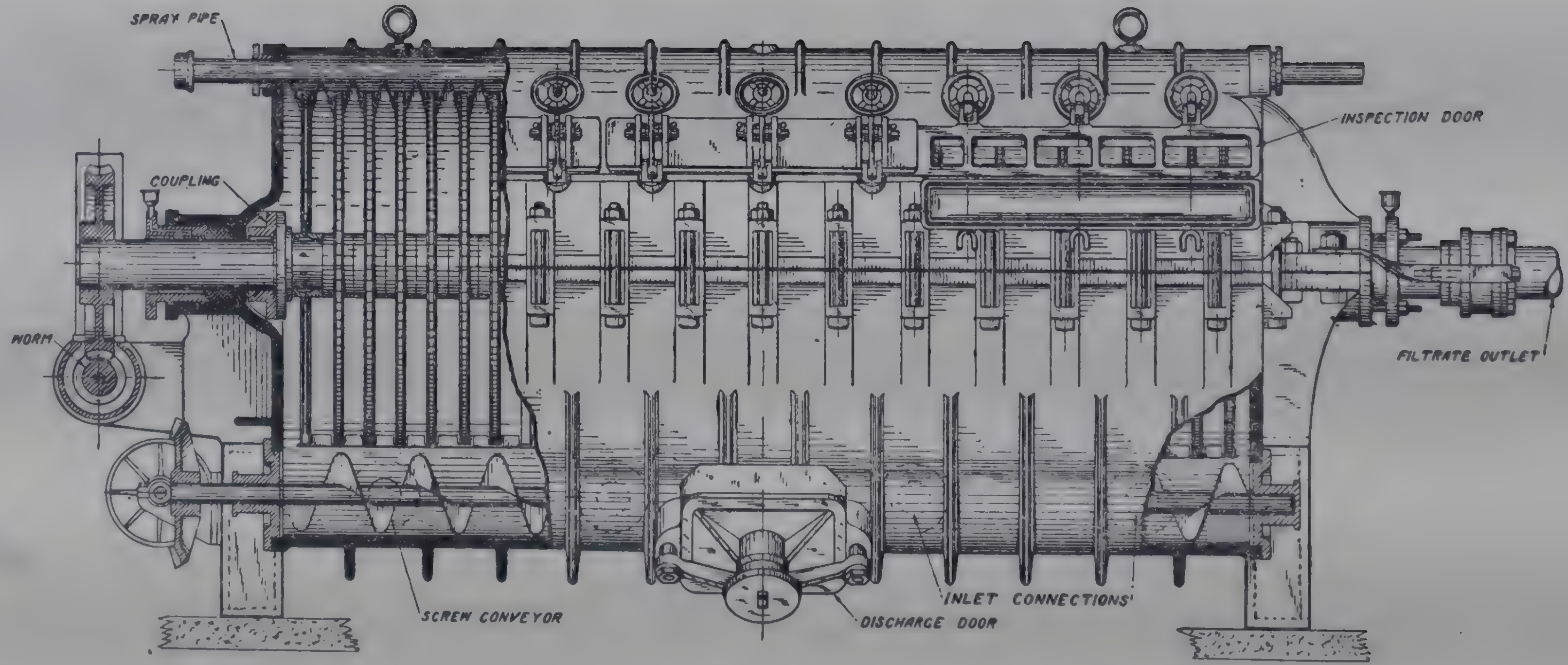
SPRAY PIPE
COUPLING
WORM
INSPECTION DOOR
FILTRATE OUTLET
SCREW CONVEYOR
DISCHARGE DOOR
INLET CONNECTIONS

Fig. 12.19. Vallez filter

The cake is discharged by the application of compressed air alone, and falls to the bottom of the shell of the filter. The solid is then moved to the outlet, situated in the centre of the bottom of the press, by means of two screw conveyors. This outlet is of adequate size to allow free discharge of the solid.

The Vallez filter produces an even cake, even with slurries that tend to sediment, and rapid handling is achieved since the press is not opened. The mechanical complications add to the cost of the plant initially and to the maintenance cost, and repairs are difficult because of the inaccessibility of the moving parts.

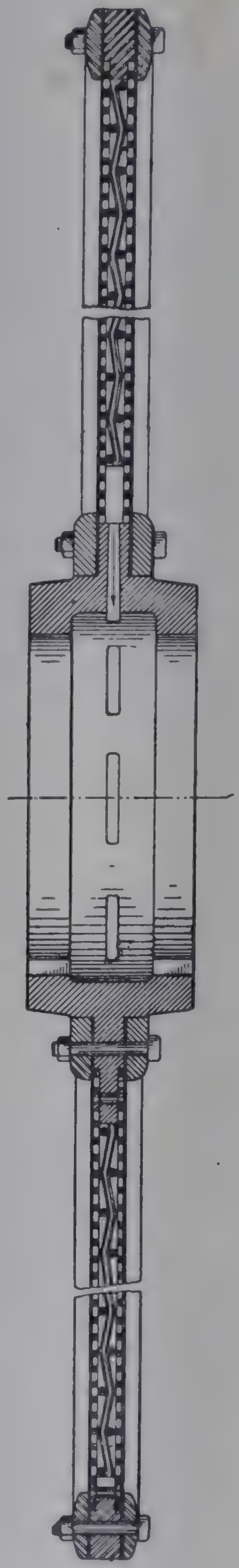

Fig. 12.20.
Leaf of a Vallez filter

### Continuous Rotary Filters

The two main types of continuous rotary filters are the rotating drum and the rotating disc filters. These are suction filters in which filtration, washing, partial drying, and discharge of the cake all take place automatically. Labour requirements are therefore low, and very economical operation can be obtained, especially if a thick suspension is fed to the filter. Rotary filters were developed for use in the mining industry, but are now widely used in the chemical industry where calcium carbonate and ammonium sulphate crystals, for example, are filtered on a large scale.

***Drum Filters.*** The rotary drum filter (Fig. 12.21) consists of a cast cylinder (Fig. 12.22) mounted horizontally, with the outer surfaces formed of perforated plate or special drainage members over which the filter cloth is fixed. The cloth is sometimes separated from the drum by a coarse metal gauze, so that the effective area for filtration is a maximum, and is held in position with wires or with a light metal screen which also protects it from damage. In all but the earliest models of drum filter, the cylinder is divided into a number of sectors and a separate connection is made between each sector and a special rotary valve. The drum is immersed to the required depth in the slurry, which is agitated to prevent settling of the solids, and vacuum is applied to those sectors of the drum which are submerged. A cake of the desired thickness, up to about 4 in., is produced by adjusting the speed of rotation of the drum. Increase of speed results in the formation of a thinner cake and consequently a higher rate of filtration. The capacity usually lies between 100 and 4000 pounds of solid per square foot per hour, and varies with the nature of the cake.

The filtration takes place at approximately constant pressure, except in the initial stages when the sector is being evacuated. This initial phase occupies

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about 3% of the filtration time for the slow filtering materials and up to 20% for the free filtering materials.

Each sector is immersed in turn in the slurry and the cake is then washed and partially dried by means of a current of air. Finally, pressure is applied under the cloth to aid the removal of the cake. The special valve consists of three main components as follows, the first two rotating with the drum and the third remaining stationary.

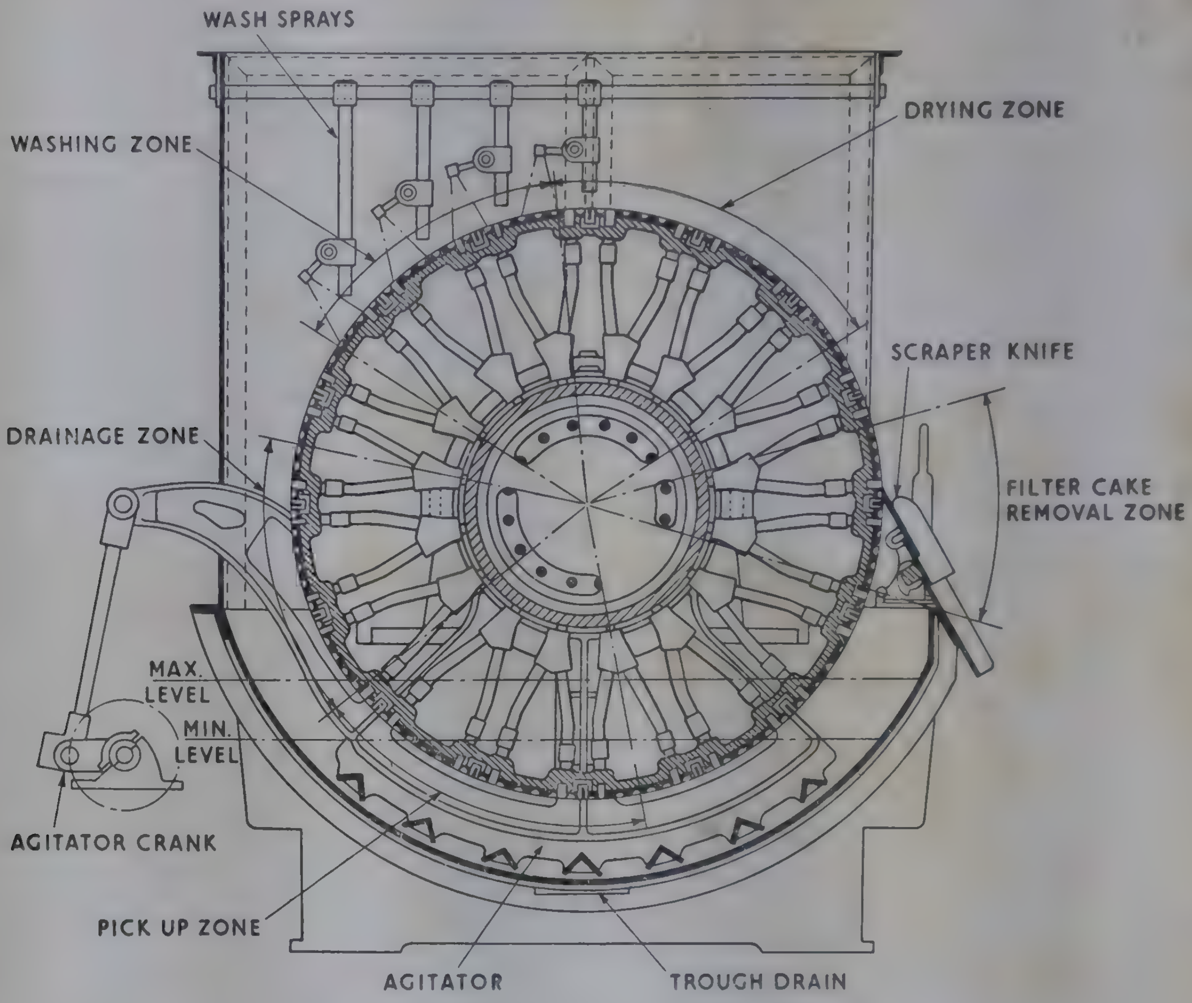


Fig. 12.25. Section through end-flow filter

(1) The valve seat which is a flat disc with ports connecting to the sectors of the filter. It can be seen in Fig. 12.22.

(2) The removable wear plate, which has holes corresponding with those on the valve seat and rotates in contact with it.

(3) The valve head, shown in Fig. 12.23, has an annular slot which connects with the filtrate outlet, the wash liquid outlet, the vacuum supply for drying the cake, and the compressed air for loosening the cake, so that each sector of the filter is connected to these in turn. A movable bridge in the slot enables the fraction of the cycle occupied by filtering and washing to be varied. Commonly, one-third of the cycle is used for filtration, one-half for washing and air drying, and the remaining sixth for the removal of the cake.

In the end-flow filter, a slightly modified form, the vacuum is applied only to sections around the periphery of the drum. (See Figs 12.24 and 12.25.) The filtrate is removed at one end of the filter through pipes which connect to a

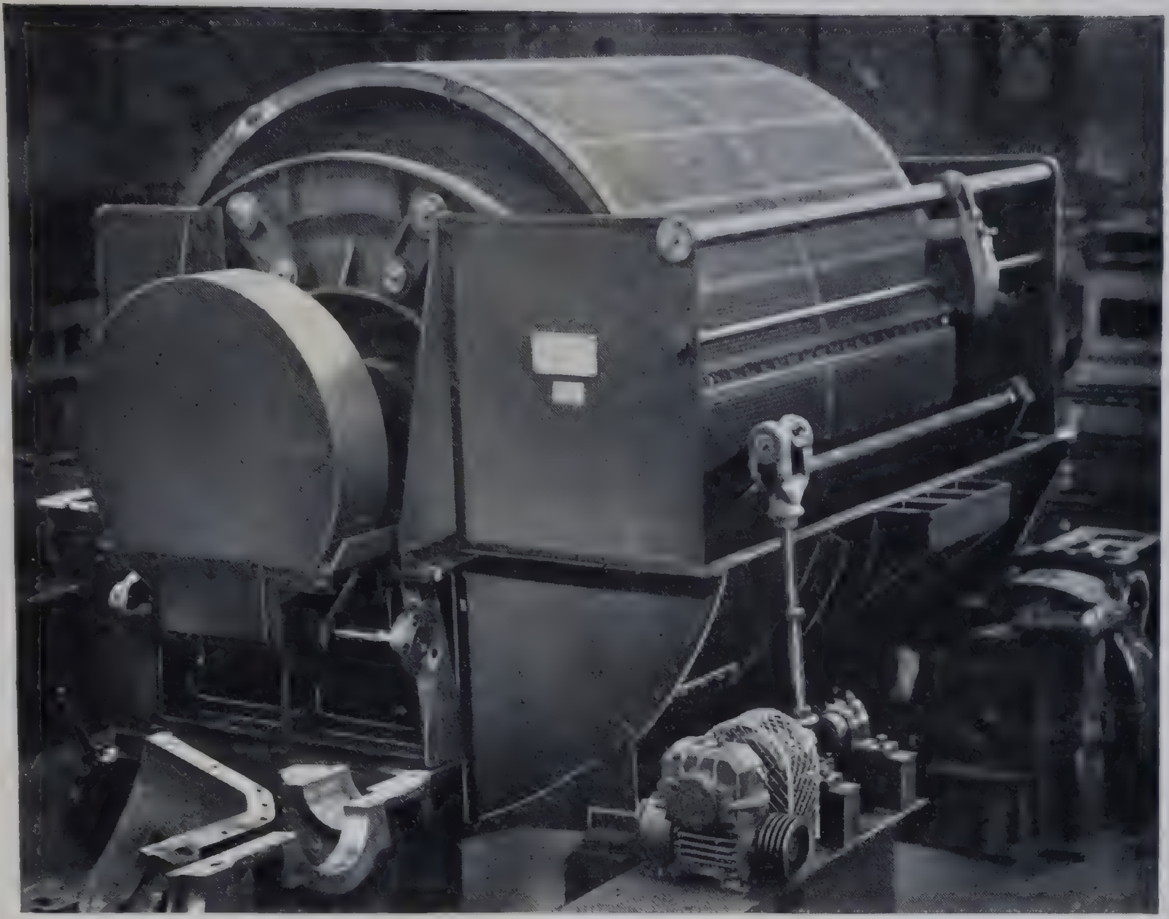


Fig. 12.21. Rotary drum filter

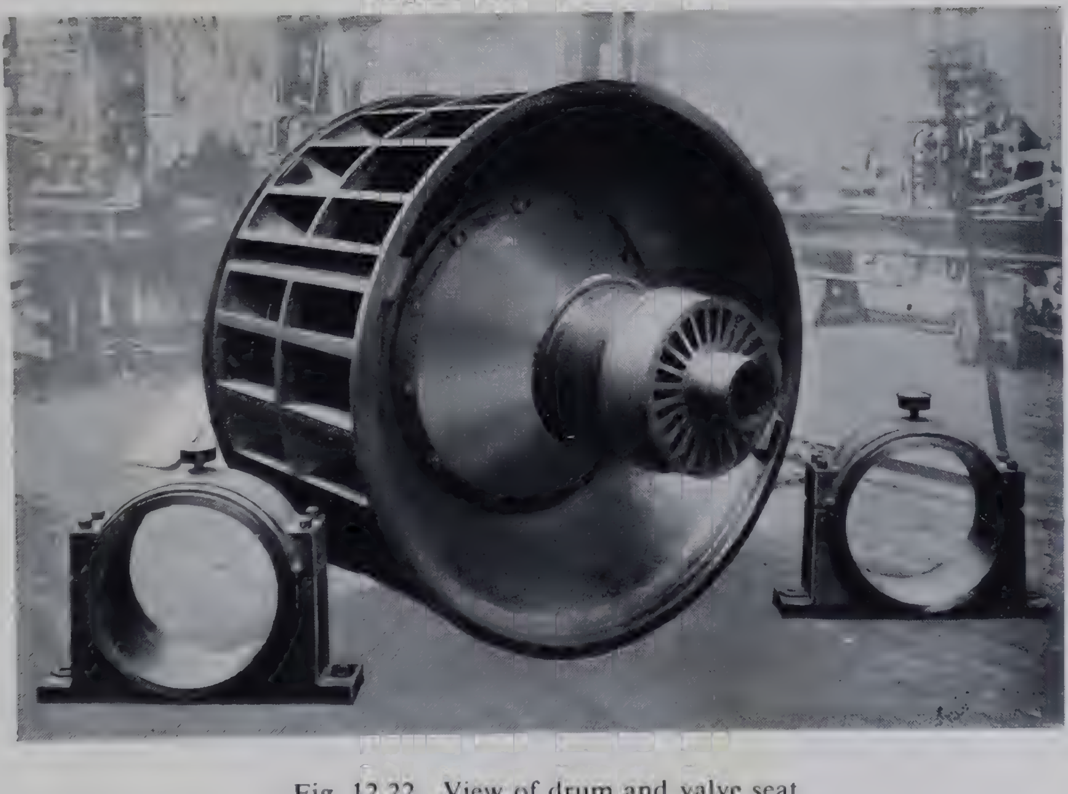


Fig. 12.22. View of drum and valve seat

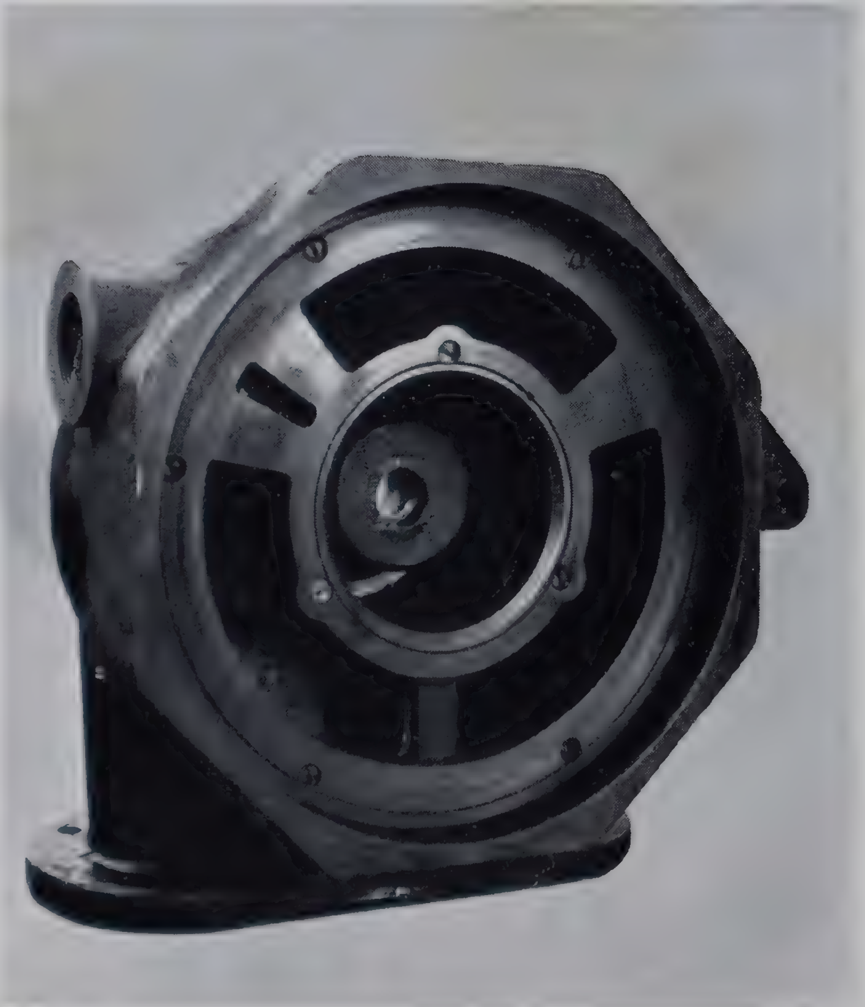
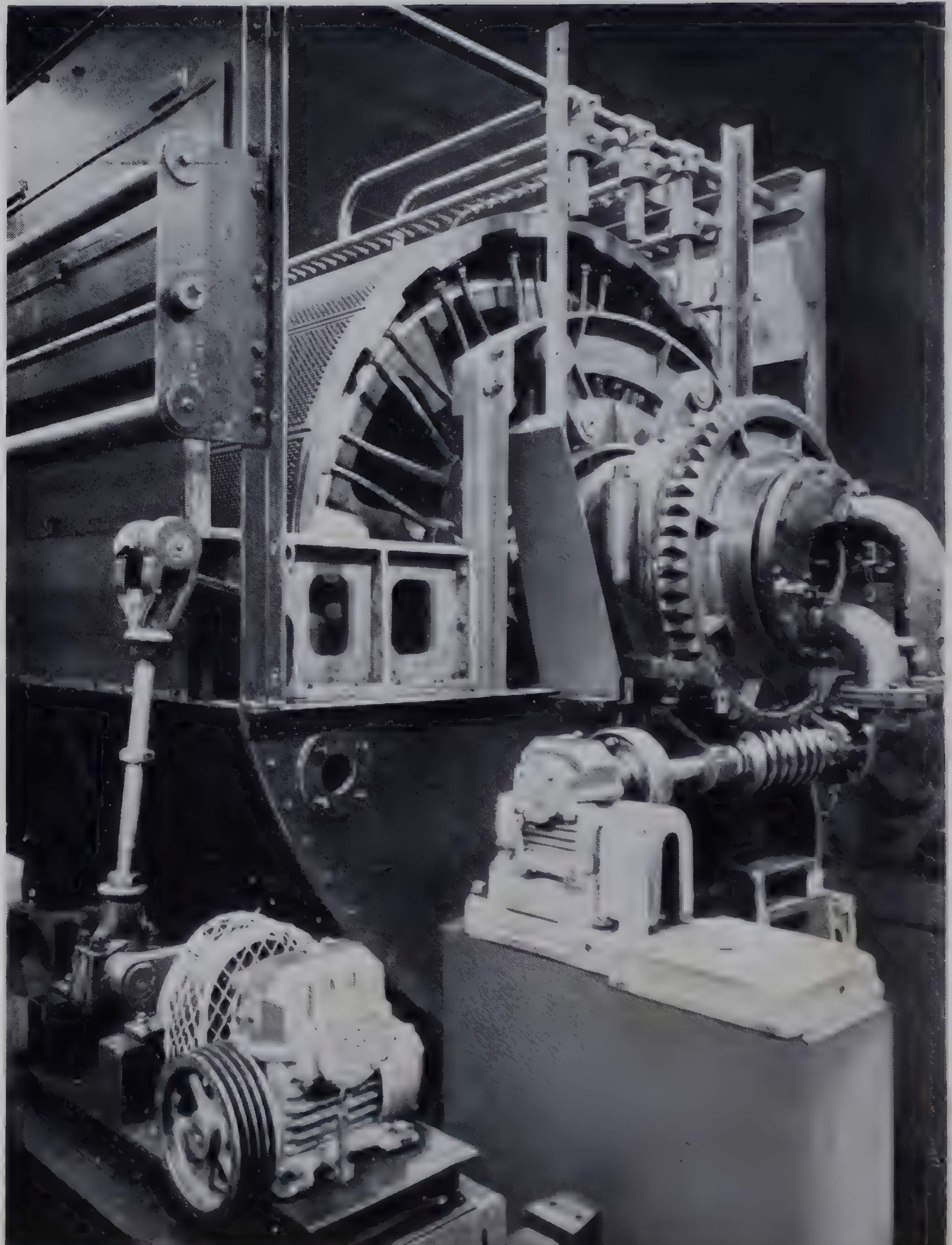


Fig. 12.23. Valve head, showing movable bridge



rotary valve similar to that already described. The advantages of this filter are that smaller stresses are set up; and the parts are more readily accessible for cleaning because the cloth and the surface of the drum can be removed in sections. It is not suitable, however, for high rates of flow of filtrate because of the resistance of the connecting pipes.

Washing is usually carried out by spraying the filter cake after it leaves the slurry, and the wash liquid then leaves the filter through the rotary valve and is collected separately from the filtrate. The sprays can be seen in Fig 12.25. If the cake cracks badly as it leaves the slurry, it may be consolidated by means of a cake compressor which consists of an endless canvas belt (Fig. 12.26) forced against the drum by means of two heavy rollers and driven by friction with the cake so that no slip occurs. The wash liquid is applied to the top of the drum and percolates through the belt and any excess runs off and is collected. The cake compressor can also be used without the application of liquid in order to obtain a comparatively dry cake. When washing is very difficult, the unwashed cake may be discharged and then mixed with wash liquid to form a fresh slurry which is then refiltered.

The capacity of the vacuum pump will be determined very largely by the amount of air sucked through the filter cake during the washing and air-drying periods when, in most cases, there will be a simultaneous flow of both liquid and air. It is not possible to calculate with any accuracy the rate of passage of air under these circumstances and the capacity of the vacuum system must be determined as a result of test experiments. A typical layout of an installation is shown in Fig 12.27, from which it is seen that the air and liquid are removed separately.

There are several methods of removing the filter cake from the drum but it is usual to employ an adjustable doctor knife (Fig. 12.25). The discharge is facilitated by the application of compressed air or steam to the under side of the cloth as this loosens the cake and raises the filter cloth slightly, bringing it into close contact with the knife. Careful adjustment of the doctor knife is essential, for if it is set too close to the drum the cloth may be damaged.

When the cake is difficult to remove, or when it is desirable to deliver it intact, a string discharge is employed. A series of closely spaced strings (Fig 12.28) pass round the drum, and the filter cake is formed over the strings which become embedded. At the discharge point the strings leave the drum and pass over two rollers and the filter cake is then released from the strings. An American company has developed a combined filtering and drying plant. A continuous belt, rather like a bed-spring in construction, passes round the under side of the drum filter and the filter cake is deposited on the belt to which it adheres. The belt leaves the filter towards the top and is then carried through a cabinet dryer. It then returns to the under side of the drum filter after the cake has been removed by agitation. The metal belt, which assists the drying of the material by virtue of its good heat-conducting properties, is formed into loops which are carried through the dryer on a slat conveyor. With finely divided materials the total loss of solid from the belt is as little as 1 or 2%.

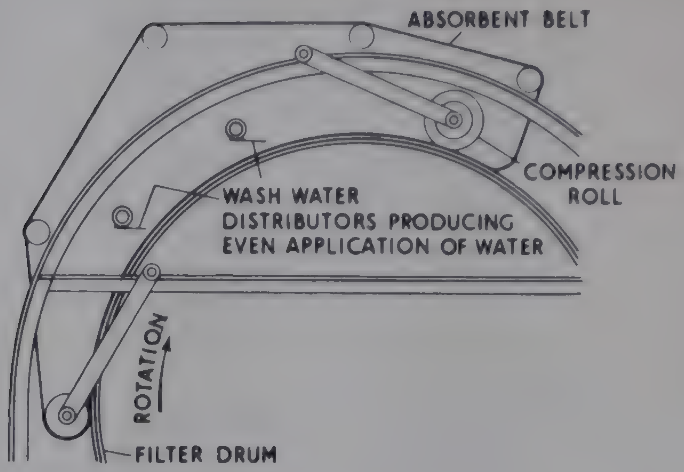

ABSORBENT BELT
COMPRESSION ROLL
WASH WATER DISTRIBUTORS PRODUCING EVEN APPLICATION OF WATER
ROTATION
FILTER DRUM

Fig. 12.26. Cake compressor

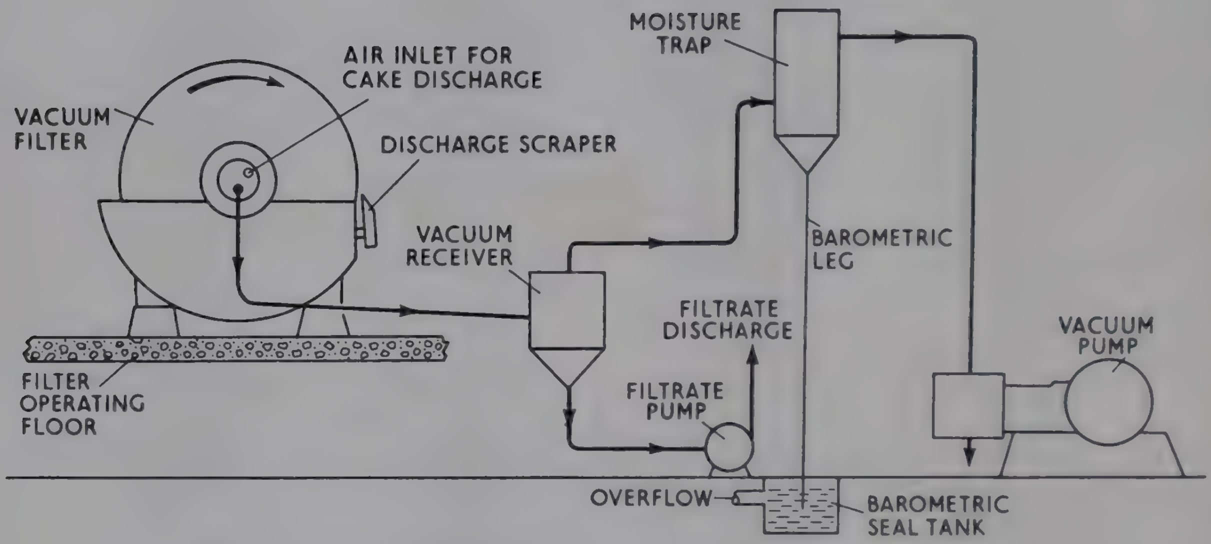

MOISTURE TRAP
AIR INLET FOR CAKE DISCHARGE
VACUUM FILTER
DISCHARGE SCRAPER
VACUUM RECEIVER
BAROMETRIC LEG
FILTRATE DISCHARGE
VACUUM PUMP
FILTER OPERATING FLOOR
FILTRATE PUMP
OVERFLOW
BAROMETRIC SEAL TANK

Fig. 12.27. Typical layout of rotary drum filter installation

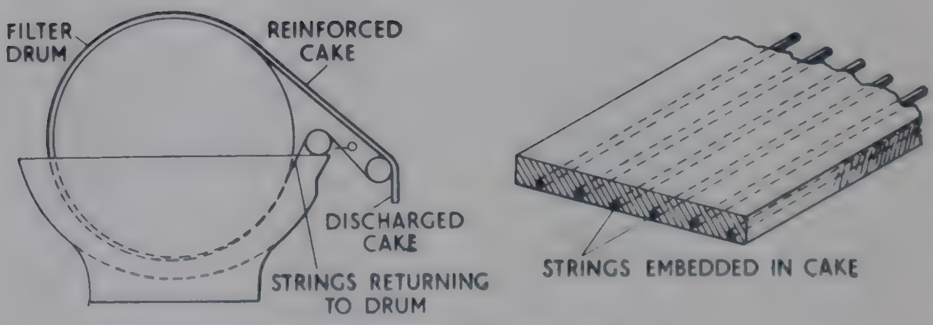

FILTER DRUM
REINFORCED CAKE
DISCHARGED CAKE
STRINGS RETURNING TO DRUM
STRINGS EMBEDDED IN CAKE

Fig. 12.28. String discharge

The slurry is agitated sufficiently to prevent settling of the particles (see agitator in Fig. 12.25), but with materials that settle very rapidly, for example slurries of coal dust or suspensions of crystals in their mother liquors, it is more satisfactory to apply the slurry from a trough at the top of the filter drum (Fig. 12.29). Because the larger particles settle more rapidly they are deposited near the filter cloth and the smaller particles form the outer portions of the filter cake. A cake of relatively high porosity is thereby obtained and high filtration rates are achieved.

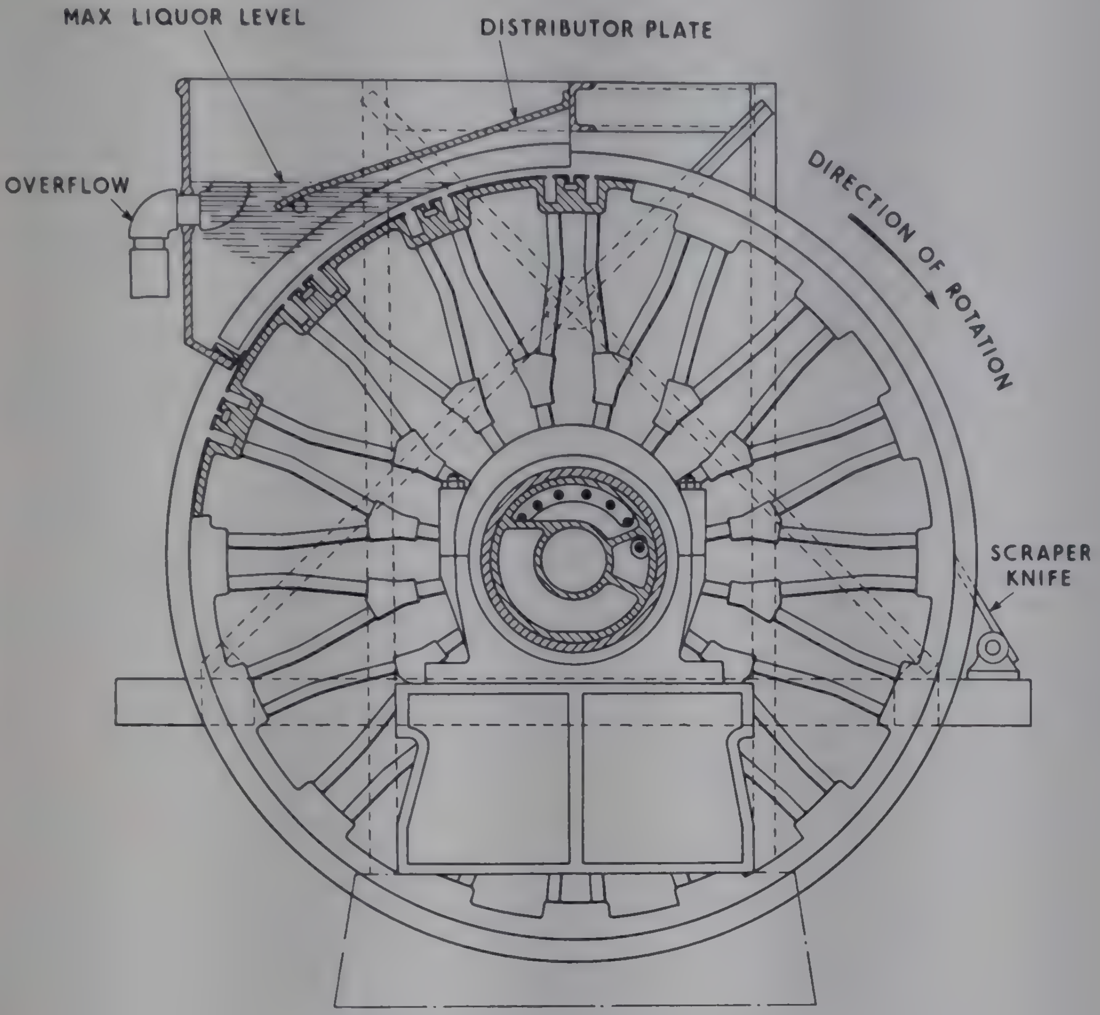


Fig. 12.29. Top feed end-flow filter

*The Internal Drum Filter*

This is a rotary drum filter (Fig. 12.30) in which filtration takes place at the inner surface of the drum; it is used with rapidly settling suspensions. The filtering surface is divided into segments in the same way as on the ordinary drum filter and each segment is connected to a separate port on the rotary valve. The slurry is fed into the drum and filtration takes place at the bottom part of its surface. The filter cake is dislodged by the application of positive pressure and vacuum alternately behind the filter cloth in the top position; this is effected automatically by means of a special rotary valve. The solid is then discharged either down a chute or on to a conveyor.

### *The Precoat Filter*

When the material forms a cake of high resistance, the process may be facilitated by the use of a precoat filter. A thick layer of a very free filtering material such as kieselguhr or asbestos is first built up on the filtering surface. Filtration is then carried out through this layer and the doctor blade is so arranged that it automatically progresses towards the drum. A very thin cake can then be formed and the whole of it removed together with a thin layer of the precoat material.

### *Advantages of the Rotary Drum Filter*

(1) The filter is entirely automatic in action and the manpower requirements are therefore reduced to a minimum.

(2) Cakes of any thickness can be built up by altering the speed of rotation of the filter. The usual range of thicknesses is from $\frac{1}{8}$ in. with fine solids to as much as 4 in. with very coarse solids. In special cases, cakes will be made as thin as $\frac{1}{16}$ in. and as thick as 12 in.

(3) The filter has a very large capacity for its size, and is extensively used for the filtration of large quantities of free filtering material.

(4) If the cake consists of coarse solids, most of the liquid can be removed from it before discharging.

### *Disadvantages*

(1) It is a vacuum filter and therefore the maximum available pressure difference is limited and difficulty is encountered in the filtration of hot liquids because of their tendency to boil.

(2) The filter cannot be used for materials that form relatively impermeable filter cakes or cakes that are difficult to remove from the cloth. The filtering properties of the slurry may be improved however by the use of filter aids.

(3) Good washing is not easily obtained, but double filtering will frequently assist.

(4) It is difficult to obtain a dry cake.

(5) The capital cost of the filter and vacuum equipment is high.

***The Rotary Disc Filter.*** The rotary disc filter (Fig. 12.31) consists of a number of circular filter leaves mounted on a heavy horizontal tubular shaft and spaced by means of hubs. The construction of the leaves can be seen in Fig. 12.32. Each sector of the disc is connected to a separate outlet and a continuous channel is formed by the outlets from the corresponding sectors of the other leaves when the discs are bolted together. These channels connect to a rotary valve, similar to that on the drum filter.

The operation of the rotary disc filter is very similar to that of the drum filter, but it gives poor washing and the discharge of the cake is more difficult. It can be operated so that several slurries are filtered simultaneously by arranging for the discs to dip into a number of different tanks. In this case it is not possible to separate the various filtrates, however. Its main advantage over the drum filter is that it gives a very much larger filtering area in the same floor space.

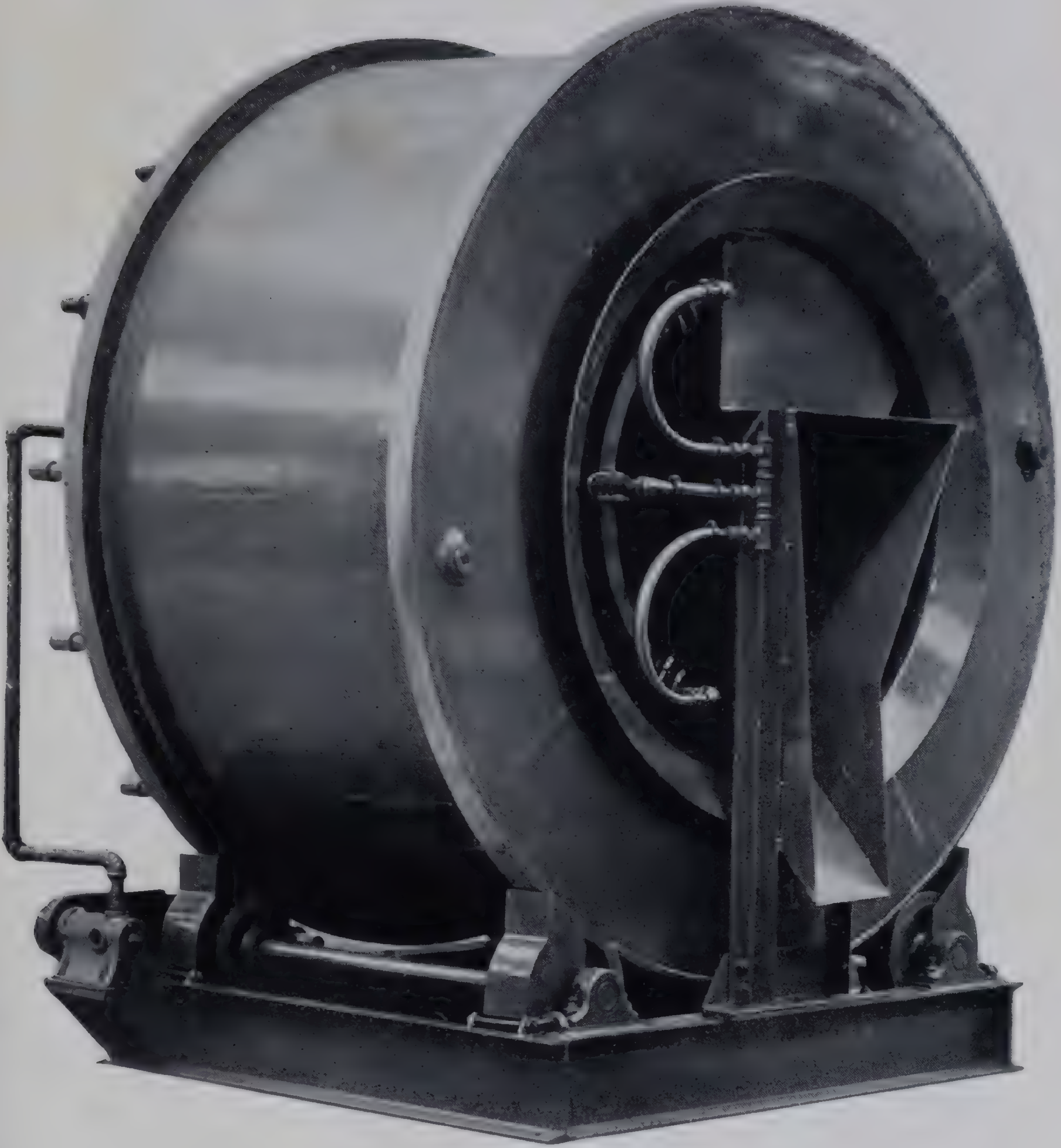


Fig. 12.30. "Dorrco" internal drum filter

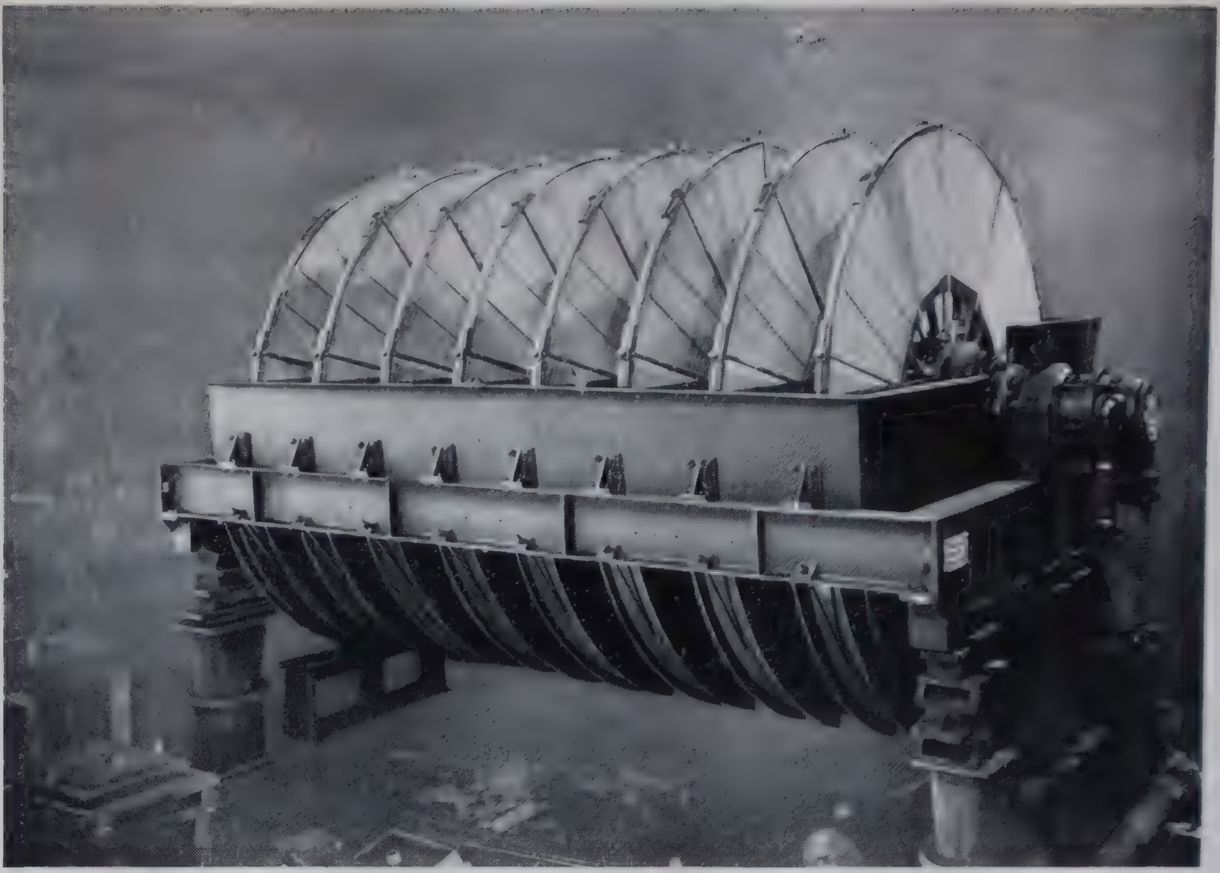


Fig. 12.31. Rotary disc filter

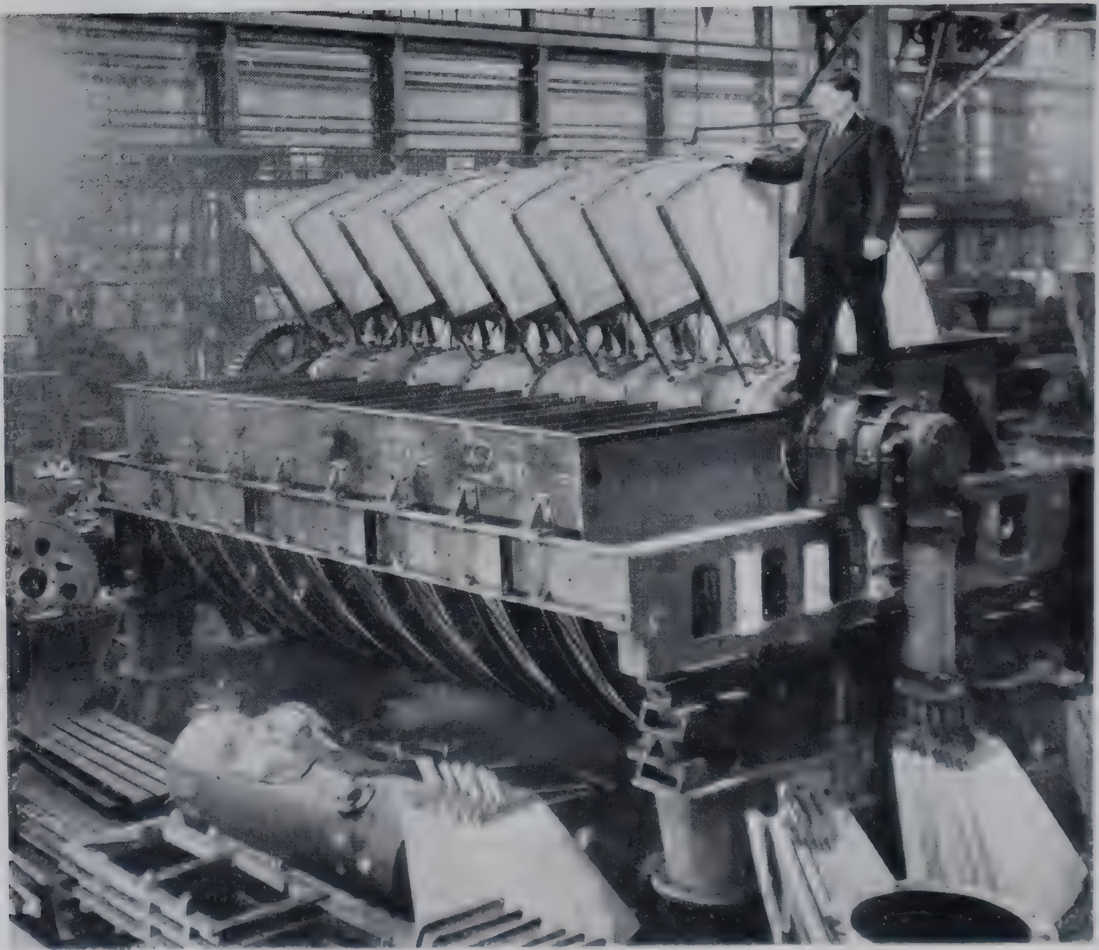


Fig. 12.32. Rotary disc filter showing construction of leaves

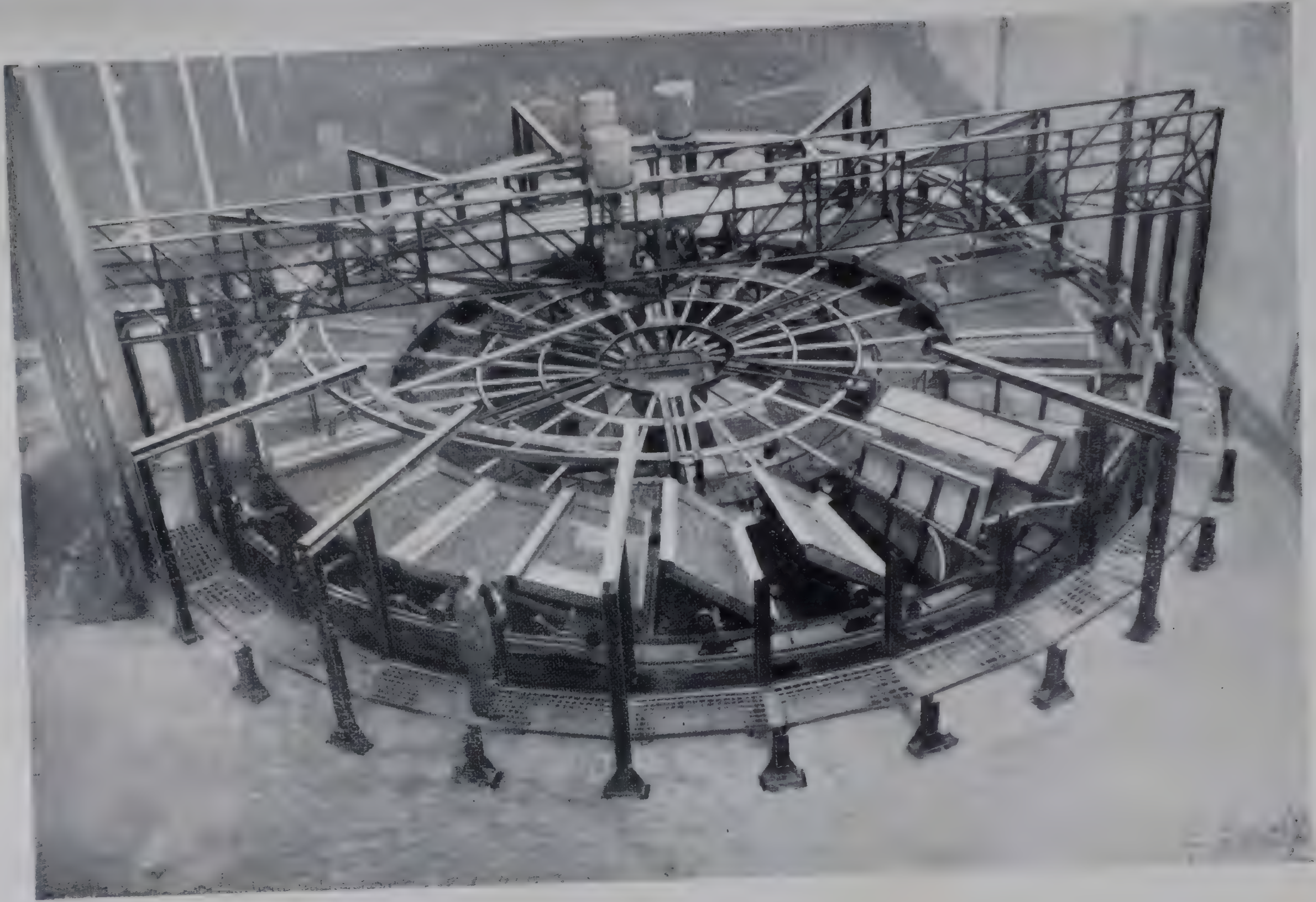
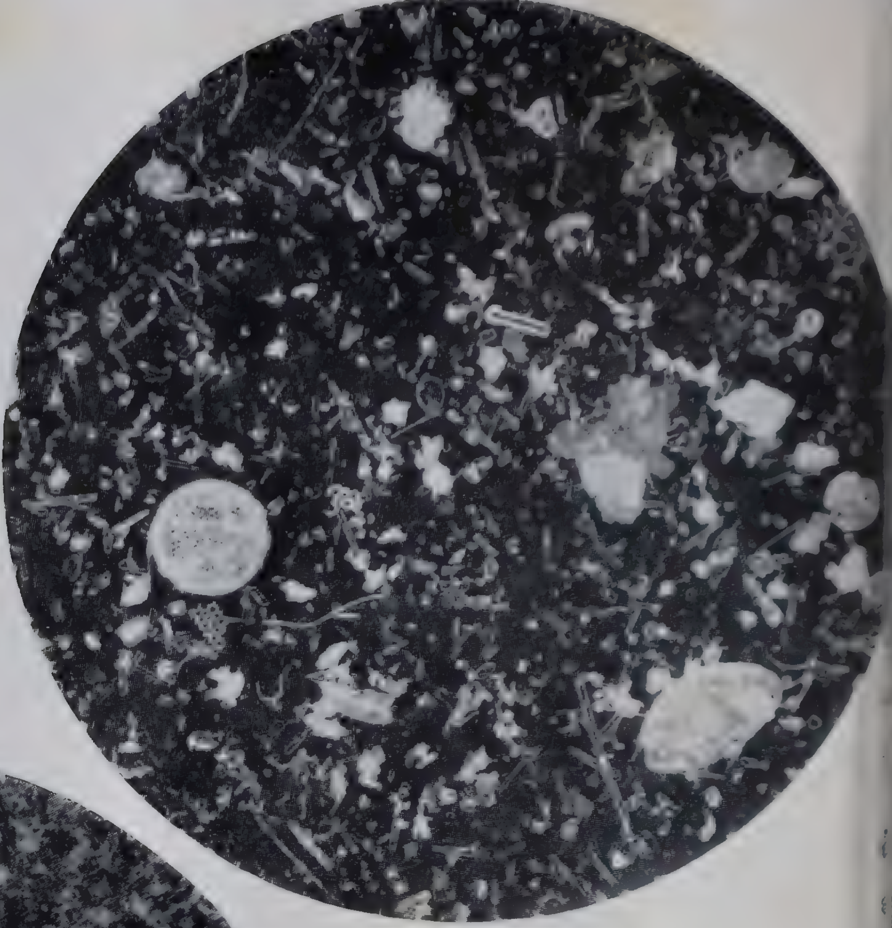
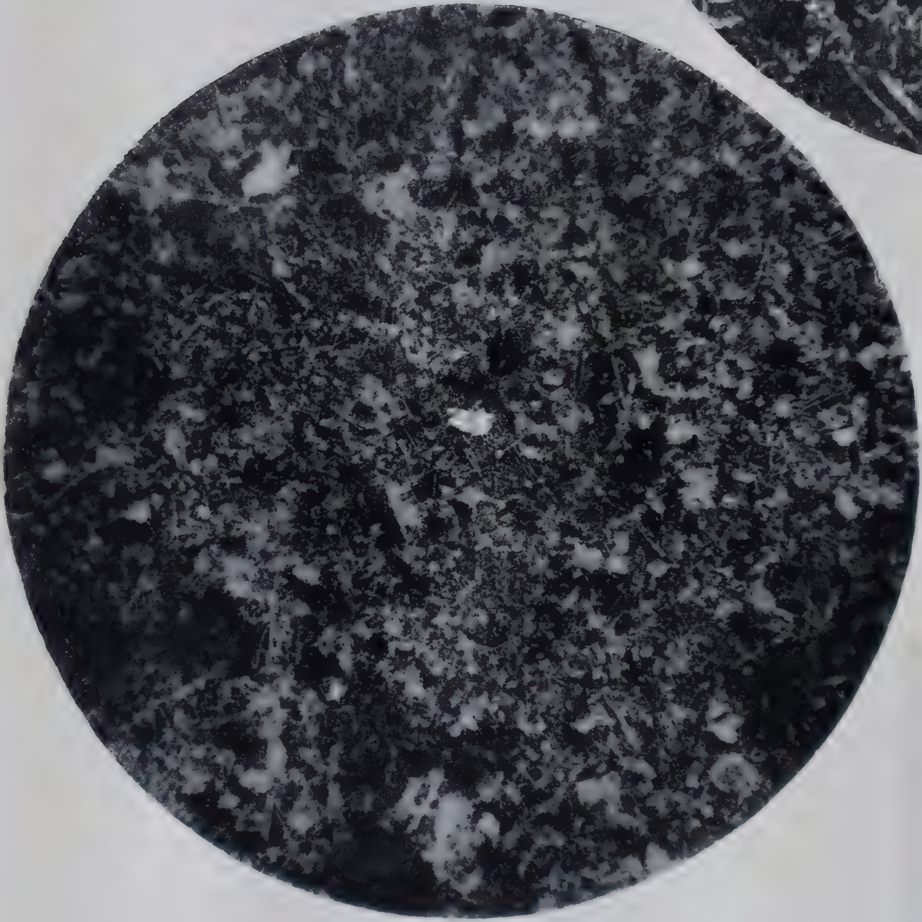


Fig. 12.33. Prayon filter



(*a*)

Fig. 12.34. Kieselguhr used for filter beds



(*b*)

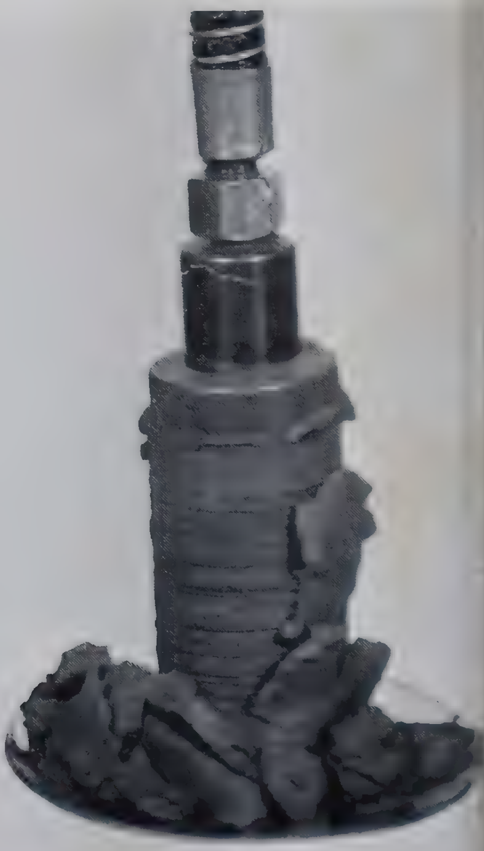


Fig. 12.35. Streamline filter pack

### The Landskrona Band Filter

Another form of completely automatic continuous vacuum filter is the Landskrona band filter⁽³⁾. A permeable belt passes over two pulleys arranged at the same vertical height. The slurry is fed on to the upper side of the belt at one end, and the filtrate is collected underneath. The speed of the belt (about 10 ft/min) and the distance over which the slurry is applied are so adjusted as to produce a cake of a suitable thickness. The cake is then subjected to the action of a spray of wash water and finally to air drying; the solid is discharged as the belt passes over the end pulley.

This filter is not extensively used in this country, but in Sweden, where it was developed, it is used for filtering phosphoric acid liquors from sludges containing calcium sulphate. It gives a very uniform filter cake free from cracks; washing is therefore good. The cost per unit area is greater than for the rotary filter.

### Prayon Continuous Filter

This filter has been developed in Belgium and Luxemburg, particularly for the vacuum filtration of the highly corrosive liquids formed in the production of phosphoric acid and phosphates, and manufacture is to begin in this country in the very near future. It is made in five sizes providing filtering surfaces from about 20 up to 430 sq. ft and the largest model is illustrated in Fig. 12.33. It consists of a number of horizontal cells held in a rotating frame, and each cell passes in turn under a slurry feed pipe and a series of wash liquor inlets. In the model illustrated, the connections are arranged so as to provide three countercurrent washes. The timing of the cycle is regulated to give the required times for filtration, washing and cake drying. The discharge of the cake is by gravity as the cell is automatically turned upside down, and can be facilitated if necessary by washing or the application of back pressure. The cloth is cleaned by spraying with wash liquid and is allowed to drain before it reaches the feed point again.

The filter has a number of advantages including:

(1) A large filtration area, up to 85% of which is effective at any instant.
(2) Low operating and maintenance costs and simple cloth replacement.
(3) Clean discharge of solids.
(4) Fabrication in a wide range of corrosion-resistant materials.
(5) Countercurrent washing.
(6) Operation at a vacuum of up to 650 mm mercury.

### The Metafilter

The Metafilter, which employs a filter bed deposited on a base of rings mounted on a fluted rod, is extensively used for clarifying liquids containing small quantities of very fine suspended solids. The rings are accurately pressed from sheet metal of very uniform thickness and are made in a large number of corrosion resistant metals, though stainless steels are usually employed. The standard rings are $\frac{7}{8}$ in. in external diameter, $\frac{5}{8}$ in. in internal diameter and 0·032 in. thick, and are scalloped on one side, as shown in Fig. 12.36, so that the edges of the discs are separated by a distance of between 0·001 and 0·010 in. according to requirements. The pack is formed by mounting the rings, all the same way up, on the drainage rod and tightening them together by a nut at one end against a boss at the other (Fig 12.37). The packs are mounted in the body of the filter which is operated either by positive or by reduced pressure.

The bed is formed by feeding to the filter a dilute suspension of material, usually a form of kieselguhr, which is strained by the packs to form a bed about $\frac{1}{8}$ in. thick. Kieselguhr is available in a number of grades and forms a bed of loose structure which is capable of trapping particles much smaller than the channels. In Fig. 12.34*a* is shown a photomicrograph of kieselguhr, and Fig. 12.34*b* gives some indication of how the particles interlock in the bed. During filtration the solids build up mainly on the surface and do not generally penetrate more than 0·02 in. into the bed. The filtrate passes between the discs and leaves through the fluted drainage rod, and operation is continued until the resistance becomes too high. The filter is then cleaned by backflushing; this causes the filter cake to crack and peel away. In some cases the cleaning may be incomplete as a result of channelling. If for any reason the spaces between the rings become blocked, the rings may be quickly removed and washed.

The Metafilter is widely used for filtering domestic water, beer, organic solvents and oils. The filtration characteristics of clay-like materials can often be improved by the continuous introduction of a small quantity of filter aid to the slurry as it enters the filter. On the other hand, when the suspended solid is coarse, the Metafilter will operate successfully as a strainer, without the use of a filter bed.

The Metafilter is very robust and is economical in use because there is no filter cloth and the bed is easily replaced. Labour charges are low. Mono pumps or diaphragm pumps are most commonly used for feeding the filter; these have already been discussed in Chapter 5.

An entirely different form of filter is available for lubricating oils (Fig. 12.38). In this, leaves or "pockets" are used in which a pair of filter papers, 4 in. in diameter and united at the edges, enclose a drainage support made up of two thin perforated metal discs, of which the inner surfaces are kept out of contact by burrs resulting from the piercing. A $\frac{5}{8}$ in. diameter hole is provided through the centre of the pockets which are mounted on a drainage pipe. Filtration takes place over the whole surface of the papers, and these are so well supported that very high pressures can be applied, even 200 lb/sq in., without causing the paper to break down. The filtration is very fine, so that fine suspended carbon can be cleared from crankcase oil, and very large areas of filter surface can be obtained in a small volume, as much as 2 sq ft of filter surface in a cylinder 4 in. diameter and 1 in. deep.

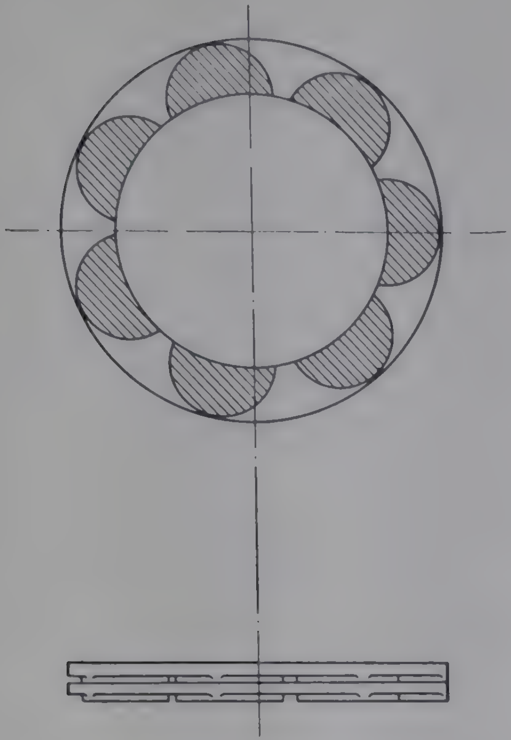


Fig. 12.36. Rings for Metafilter

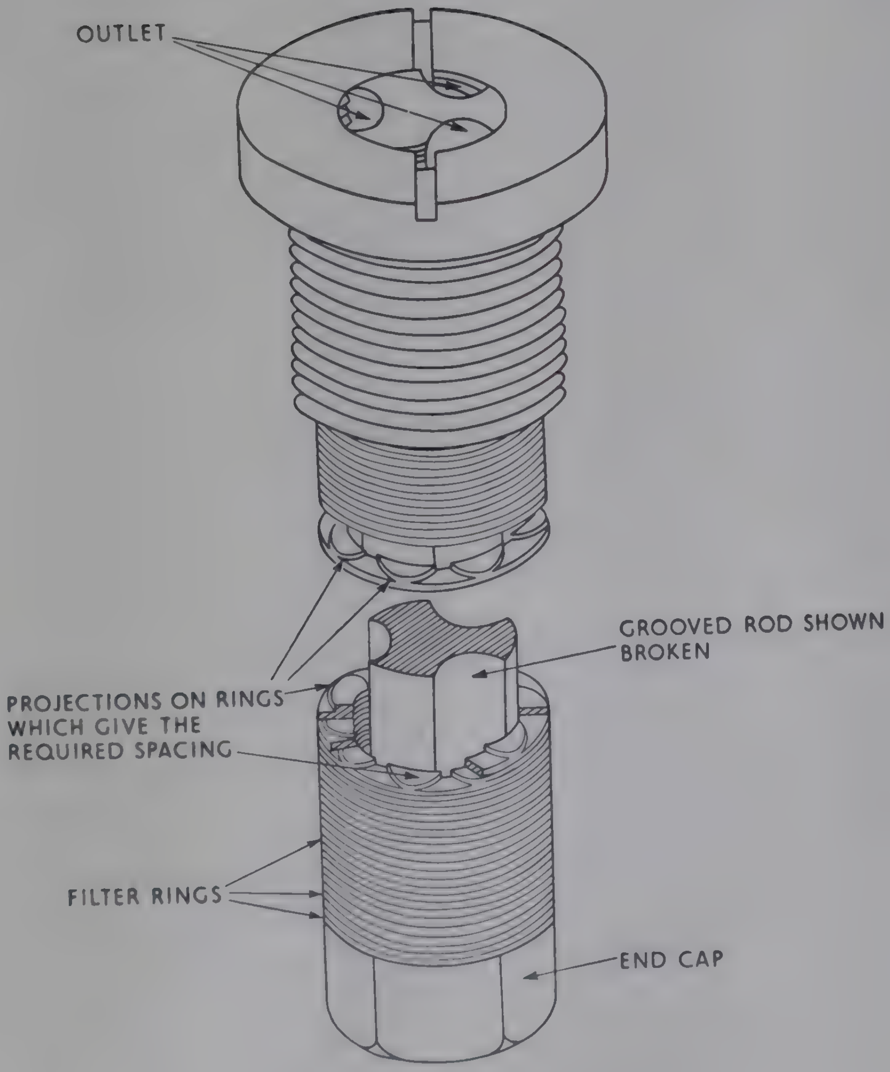

OUTLET
GROOVED ROD SHOWN BROKEN
PROJECTIONS ON RINGS WHICH GIVE THE REQUIRED SPACING
FILTER RINGS
END CAP

Fig. 12.37. Metafilter pack

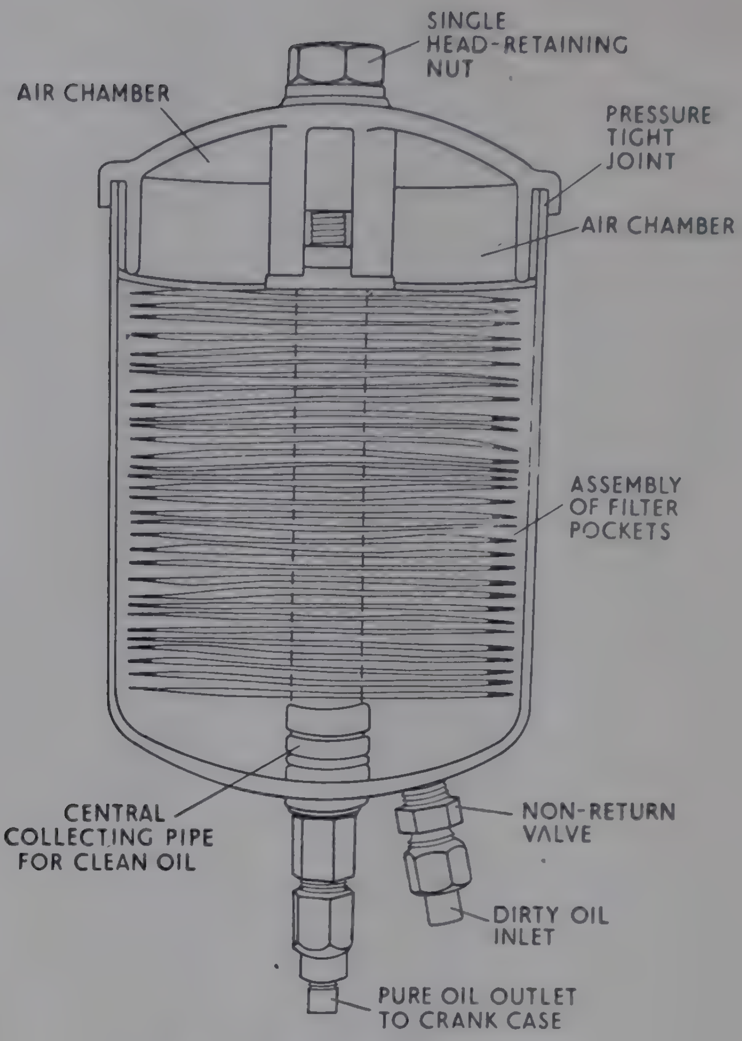

SINGLE HEAD-RETAINING NUT
AIR CHAMBER
PRESSURE TIGHT JOINT
AIR CHAMBER
ASSEMBLY OF FILTER POCKETS
CENTRAL COLLECTING PIPE FOR CLEAN OIL
NON-RETURN VALVE
DIRTY OIL INLET
PURE OIL OUTLET TO CRANK CASE

Fig. 12.38. Metafilter oil filter

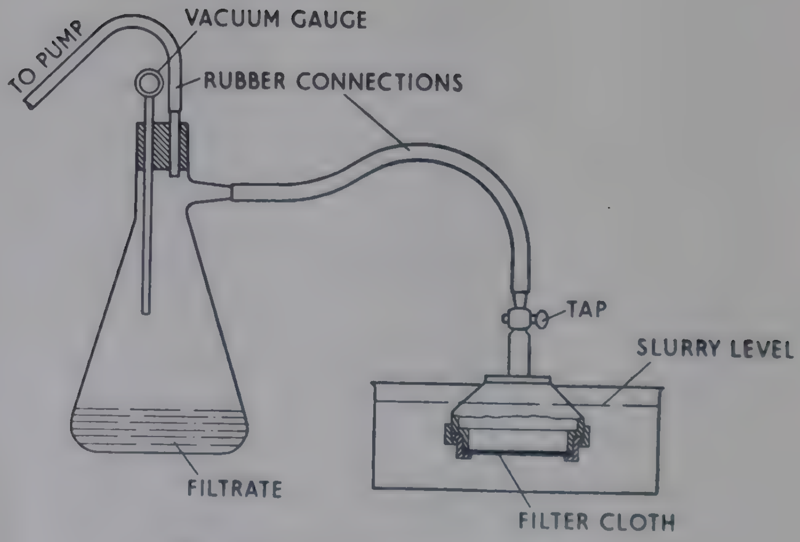

TO PUMP
VACUUM GAUGE
RUBBER CONNECTIONS
TAP
SLURRY LEVEL
FILTRATE
FILTER CLOTH

Fig. 12.39. Laboratory test filter

## The Streamline Filter

The Streamline filter is geometrically similar to the Metafilter, but the cylindrical filter packs (Fig. 12.35) are formed from compressed paper discs about 2 in. in diameter. No filter bed is required and the liquid flows radially inwards through the small spaces between the individual papers and through the papers themselves. The filter pack is considerably cheaper than that in the Metafilter, but it needs replacing at intervals and the same pack cannot be used for filtering a number of different liquids. The Streamline filter is extensively used as a petrol filter and also for oils; it does not operate successfully with aqueous liquids.

## Selection of a Filter

Whereas it may be possible to predict qualitatively the effect of the physical properties of the fluid and the solid on the filtration characteristics of a suspension, it is necessary in all cases to carry out a test on a sample before the large-scale plant can be designed. A simple vacuum filter which is used to obtain laboratory data is illustrated in Fig. 12.39; the area of the filter should be not less than 10 sq in. The information on filtration rates and specific resistance obtained in this way can be directly applied to industrial filters provided due account is taken of the compressibility of the filter cake.

**Example.** A slurry is filtered in a plate and frame press containing 12 frames, each 1 ft square and 1 in. thick. During the first three minutes, the filtration pressure is slowly raised to the final value of 60 lb/sq in. and, during this period, the rate of filtration is maintained constant. After the initial period, filtration is carried out at constant pressure and the cakes are completely formed in a further 15 minutes. The cakes are then washed at 40 lb/sq in. for 10 minutes, using "thorough washing". What is the volume of filtrate collected per cycle and how much wash water is used?

A sample of the slurry had previously been tested, using a vacuum leaf filter of $\frac{1}{2}$ sq ft filtering surface and a vacuum of 20 in. mercury. The volume of filtrate collected in the first five minutes was 250 cc and, after a further 5 minutes, an additional 150 cc were collected. Assume the cake to be incompressible and the cloth resistance to be the same in the leaf as in the filter press.

**Solution.** In leaf filter, filtration is at constant pressure from start. Thus

$$V^2 + 2\,\frac{AL}{v}\cdot V = 2\,\frac{\Delta P A^2}{r\mu v}\cdot t \qquad \text{(from equation 12.14}a\text{)}$$

In the filter press, a volume $V_1$ of filtrate is obtained under constant rate conditions in time $t_1$, and filtration is then carried out at constant pressure.

Thus $$V_1^2 + \frac{AL}{v}\cdot V_1 = \frac{\Delta P A^2}{r\mu v}\cdot t \qquad \text{(from equation 12.13}b\text{)}$$

and $$(V^2 - V_1^2) + 2\,\frac{Al}{v}\,(V - V_1) = 2\,\frac{\Delta P A^2}{r\mu v}\,(t - t_1) \qquad \text{(from equation 12.14}a\text{)}$$

*For leaf filter—*

Work with pressures in pounds per square inch, volumes in litres, areas in square feet, time in minutes.

When $t = 5$, $V = 0{\cdot}25$ and when $t = 10$, $V = 0{\cdot}4$.

$A = \frac{1}{2}$ and $\Delta P = 9{\cdot}8$

Thus
$$0{\cdot}25^2 + 2 \,.\, \tfrac{1}{2} \,.\, L/v \,.\, 0{\cdot}25 = \frac{2 \times 9{\cdot}8 \times \frac{1}{4}}{r\mu v} \times 5 \quad \text{, and}$$

$$0{\cdot}4^2 + 2 \,.\, \tfrac{1}{2} \,.\, L/v \,.\, 0{\cdot}4 = \frac{2 \times 9.8 \times \frac{1}{4}}{r\mu v} \times 10$$

i.e.
$$0{\cdot}0625 + 0{\cdot}25\ L/v = \frac{24{\cdot}5}{r\mu v} \quad \text{, and}$$

$$0{\cdot}16 + 0{\cdot}4\ L/v = \frac{49}{r\mu v}$$

Hence
$$L/v = 0{\cdot}35 \text{ and } r\mu v = 163$$

The volume of filtrate $V_1$ collected during the constant rate period on the filter press is given by ($A = 24$, $\Delta P = 60$, and $t = 3$)—

$$V_1^2 + 24 \times 0{\cdot}35 V_1 = \frac{60 \times 576}{163} \times 3$$

i.e.
$$V_1^2 + 8{\cdot}4 V_1 - 636 = 0$$

$\therefore$
$$V_1 = -4{\cdot}2 + \sqrt{17{\cdot}7 + 636} = 21{\cdot}4 \text{ litres}$$

For the constant pressure period,
$$t - t_1 = 15 \text{ minutes.}$$

The total volume of filtrate collected is therefore given by,

$$(V^2 - 458) + 16{\cdot}8(V - 21{\cdot}4) = 424 \times 15$$

i.e.
$$V^2 + 16{\cdot}8V - 7177 = 0$$

$\therefore$
$$V = -8{\cdot}4 + \sqrt{70{\cdot}3 + 7177}$$
$$= \underline{\underline{76{\cdot}7 \text{ litres}}}$$

Final rate of filtration
$$= \frac{\Delta P A^2}{r\mu v(V + AL/v)} \qquad \text{(from equation 12.12)}$$

$$= \frac{212}{76{\cdot}7 + 24 \times 0{\cdot}35} = 2{\cdot}48 \text{ litres per minute}$$

If the viscosity of the filtrate is the same as that of the wash-water,

Rate of washing at 60 lb/sq in. = 0·62 litres/minute.
Rate of washing at 40 lb/sq in. = 0·42 litres/minute.

Thus the amount of wash-water passing in 10 minutes = $\underline{\underline{4{\cdot}2 \text{ litres.}}}$

**Example.** A slurry, containing 0·2 lb of solid (specific gravity 3·0) per pound of water, is fed to a rotary drum filter, 2 ft long and 2 ft diameter. The drum rotates at one revolution in 6 minutes and 20% of the filtering surface is in contact with the slurry at any instant. If filtrate is produced at the rate of 1000 lb per hour and the cake has a voidage of 0·5, what thickness of cake is produced when filtering with a vacuum of 20 in. of mercury?

The rotary filter breaks down and the operation has to be carried out temporarily in a plate and frame press with frames 1 ft square. The press takes 2 minutes to dismantle and 2 minutes to re-assemble and, in addition, 2 minutes are required to remove the cake from each frame. If filtration is to be carried out at the same overall rate as before, with an operating pressure of 25 lb/sq. in. gauge, what is the minimum number of frames that need to be used and what is the thickness of each? Assume the cakes to be incompressible and neglect the resistances of the filter media.

**Solution.**

*Drum Filter—*

Area of filtering surface $= 2 \times 2\pi = 4\pi$ sq. ft

Rate of filtration $= 1000$ lb/hr

$$= \frac{1000}{60 \times 62{\cdot}3} = 0{\cdot}270 \text{ cu. ft/min. of filtrate.}$$

Volumetric rate of deposition of solids (bulk)

$$= 0{\cdot}270 \times 0{\cdot}2 \times \frac{1}{0{\cdot}5} \times \frac{1}{3{\cdot}0} = 0{\cdot}270/7{\cdot}5 = 0{\cdot}036 \text{ cu. ft/min.}$$

1 revolution takes 6 minutes; therefore given piece of filtering surface is immersed for $6 \times 0{\cdot}2 = 1{\cdot}2$ min.

Bulk volume of cake deposited per revolution $= 0{\cdot}036 \times 6 = 0{\cdot}216$ cu ft

Thickness of cake produced $= \dfrac{0{\cdot}216 \times 12}{4\pi} = \underline{\underline{0{\cdot}204 \text{ in.}}}$

Now $$\frac{dV}{dt} = \frac{\Delta PA}{r\mu l} = \frac{\Delta PA^2}{r\mu Vv} \quad \text{(from equations 12.2 and 12.7)}$$

At constant pressure, $$V^2 = \frac{2}{r\mu v}\Delta PA^2 t = K\Delta PA^2 t \text{ (say)} \quad \text{(from equation 12.9a)}$$

Then expressing pressures, areas, times and volumes in lb/sq in., square feet, minutes and cubic feet, respectively, for one revolution of the drum,

$$(0{\cdot}270 \times 6)^2 = K \times 10 \times 16\pi^2 \times 1{\cdot}2$$

(since each element of area is immersed for one fifth of cycle)

i.e. $$K = 0{\cdot}00136$$

*Filter Press—*

Use a filter press with $n$ frames of thickness $d$ inches.

Total time, in minutes, for one complete cycle of press $= t_f + 2n + 4$

where $t_f$ is the time during which filtration is occurring.

$$\text{Overall rate of filtration} = \frac{V_f}{t_f + 2n + 4} = 0{\cdot}270 \text{ cu ft/min}$$

(where $V_f$ is the total volume of filtrate per cycle)

Now $V_f$ = Volume of frames/volume of cake deposited by unit volume of filtrate ($v$)
$= 7{\cdot}5n \,.\, d/12$

$$\text{But} \quad V_f^2 = 0{\cdot}00136 \times 25 \times (2n)^2 \times t_f = \frac{7{\cdot}5^2n^2d^2}{144}$$

$$\text{i.e.} \quad t_f = \frac{7{\cdot}5^2n^2d^2}{0{\cdot}00136 \times 25 \times 4n^2 \times 144} = 2{\cdot}88d^2$$

$$\text{Thus } 0{\cdot}270 = \frac{7{\cdot}5nd}{12(2{\cdot}88d^2 + 2n + 4)}$$

i.e.

$$2{\cdot}88d^2 + 2n + 4 = 2{\cdot}31nd$$

giving

$$n = \frac{4 + 2{\cdot}88d^2}{2{\cdot}31d - 2}$$

$n$ is a minimum when $\frac{dn}{dd} = 0$,

i.e. when

$$(2{\cdot}31d - 2) \times 5{\cdot}76d - (4 + 2{\cdot}88d^2) \times 2{\cdot}31 = 0$$
$$13{\cdot}3d^2 - 11{\cdot}5d - 9{\cdot}2 - 6{\cdot}66d^2 = 0$$
$$6{\cdot}6d^2 - 11{\cdot}5d - 9{\cdot}2 = 0$$
$$d^2 - 1{\cdot}73d - 1{\cdot}38 = 0$$
$$d = 0{\cdot}865 \pm \sqrt{0{\cdot}75 + 1{\cdot}38}$$
$$= 0{\cdot}865 + 1{\cdot}46$$
$$= 2{\cdot}32 \text{ inches}$$

Hence $n = 5{\cdot}7.$

Thus a minimum of 6 frames must be used.

The sizes of frames which will give exactly the required rate of filtration when six are used are given by,

$$4 + 2{\cdot}88d^2 = 13{\cdot}9d - 12$$

i.e.

$$d^2 - 4{\cdot}8d + 5{\cdot}5 = 0$$

$$\therefore \quad d = 2{\cdot}4 \pm \sqrt{5{\cdot}76 - 5{\cdot}5}$$
$$= 1{\cdot}9 \quad \text{or} \quad 2{\cdot}9 \text{ in.}$$

i.e. 6 frames of thickness 1·9 in. or 2·9 in. will give exactly the required filtration rate; intermediate sizes give higher rates.

Thus any frame thickness between 1·9 and 2·9 inches will be satisfactory. In practice, 2 or 2½ inch frames would be used.

## CHAPTER 13

# *The Centrifuge*

The centrifuge consists of a basket in which a mixture of solid and liquid, or a mixture of two liquids, is rotated at high speed so that it is separated into its constituents by the action of centrifugal force. The basket may be perforated, in which case liquid passes out through the holes, or it may be imperforate and the liquid removed through a skimming tube or over a weir. The centrifuge is frequently used for drying crystals and for separating emulsions into their constituent liquids.

A simple gravity settling process (A) and a centrifugal separation of solid particles from a liquid (B) are illustrated in Fig. 13.1. In the former case the particles settle vertically downwards, and in the latter they travel radially relative to the liquid. When sedimentation is complete, so that the particles are all supported on the bottom of the vessel, the hydrostatic pressure is attributable to the total depth $w$ of liquid, if it is assumed that there is a through passage for the fluid between the particles. The weight of the particles has no effect on the pressure because they are supported independently of the liquid. Similarly the centrifugal pressure due to the fluid is independent of the presence of the layer of particles on the walls of the basket. The difference between gravity settling and centrifugal separation of a mixture of oil and dirt and water is illustrated in Fig. 13.2, in which the left hand tube was allowed to stand for 24 hours and the right hand tube was subjected to centrifugal force for one minute only.

If a particle of mass $m$ is rotating at a radius $x$ with an angular velocity $\omega$, it is subjected to a centrifugal force $mx\omega^2$ in a radial direction, and a gravitational force $mg$ in a vertical direction, where $g$ is the acceleration due to gravity. The ratio of the centrifugal to the gravitational force, $\dfrac{x\omega^2}{g}$, is a measure of the separating power of the machine and often has a value of several thousand. Separations can therefore be carried out very much more rapidly in a centrifuge than under the action of gravity. In addition, the degree of separation which is ultimately obtainable may be very much greater because the forces available are of a far higher order of magnitude. Thus, for example, whereas relatively coarse solids can be separated from a fluid by gravitational sedimentation, very fine solids cannot be separated in this manner, because Brownian motion is sufficient to maintain an appreciable proportion of the particles in suspension. On the other hand, if the separating force is greatly increased by the use of a centrifuge, the forces giving rise to mixing become negligible in comparison, so that virtually complete separation may be obtained. A similar situation exists in the draining of mother liquor from a batch of crystals. Some of the liquid is held between the surfaces of the crystals by surface tension forces and therefore only a limited degree of separation can be achieved by draining under gravity. If centrifugal separation is employed, a far higher proportion of the liquid can be removed and a substantially dry product obtained. High speed centrifuges are therefore used for the separation of colloidal suspensions and emulsions which are stable under the action of the earth's gravitational field. This application is frequently of value for laboratory analysis.

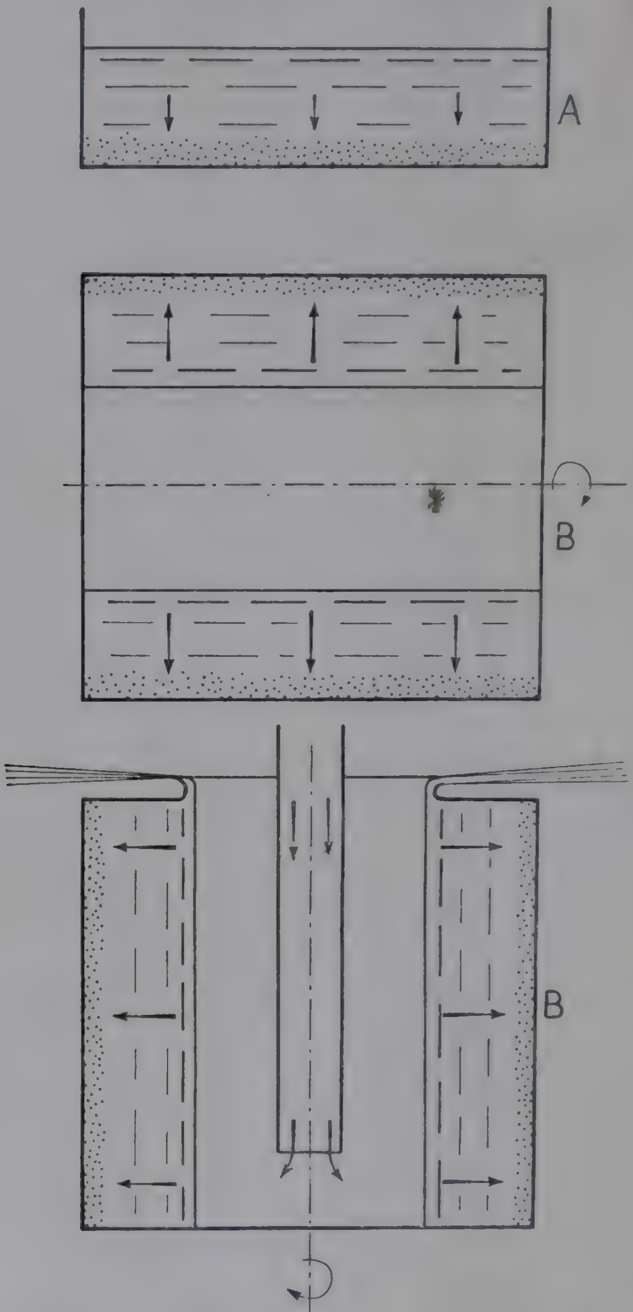


Fig. 13.1. Principle of gravity settling (A) and centrifugal separation (B)

Whereas gravitational settling has been carried out from the earliest times, centrifuges were not introduced until the middle of the nineteenth century. All the early machines operated on the batch principle and their capacity was limited because considerable time was lost in bringing the basket up to speed and in bringing it to rest again at the end of the operation. Recent trends in design have been towards the development of continuous machines which can be run at constant speed, and from which each of the separated products can be continuously discharged.

Most small batch centrifuges are mounted with their axes vertical and, because of the possibility of uneven loading of the machine, the basket is normally supported in bearings either above or below, but not in both positions, so that a certain degree of flexibility is provided. In the underdriven machine, where the drive and bearings are underneath (Fig. 13.3), access to the basket is easier and the material is normally discharged from the top. In the overdriven centrifuge (Fig. 13.4), in which the basket is suspended, a flap valve can be incorporated in the bottom of the basket for easy discharge of the solids. This latter type of machine is the more versatile and can cope more readily with uneven loading. The large continuous centrifuges usually have their axes horizontal: even conditions then prevail for the whole length of the basket and the removal of the deposited solids is easier. In some cases, the axis is inclined slightly to the horizontal as this further facilitates discharging the solids.

Centrifuges with imperforate baskets are used either for producing an accelerated separation of solid particles in a liquid, or for separating mixtures of two liquids. In the former case, the solids are deposited on the walls of the basket and the liquid is removed through an overflow or skimming tube. The suspension is continuously fed in until a suitable depth of solids has been built up on the walls; this deposit is then removed either by hand or by a mechanical scraper. When a mixture of liquids is to be separated the denser liquid collects near the walls and the lighter liquid forms an inner layer. Overflow weirs are arranged so that the two constituents are continuously removed: the design of the weirs will be considered on page 460.

Perforated baskets are used when relatively large particles are to be separated from a liquid, as for example in the separation of mother liquor and drying of crystals. The mother liquor passes through the bed of particles and then through the perforations in the basket. When the centrifuge is used for filtration, a coarse gauze is laid over the inner surface of the basket and the filter cloth rests on the gauze; space is thus provided behind the cloth for the filtrate to flow to one of the perforations. In the initial stages, the particles settle on to the cloth and the clarified liquid passes through the bed of material so deposited. If a continuous stream of slurry is maintained, the concentration of the suspension in the basket rapidly reaches an equilibrium value.

In the valve nozzle type of centrifuge, which is used in the processing of yeast, starch, meat and fish products and fruit juices, the solids are discharged through the wall of the machine under controlled conditions. The centrifuge forms an essential component of the comparatively new process of impulse rendering for the extraction of oils and fats from cellular materials. The raw material, consisting of bones, animal fat, fish offal or vegetable seeds, is first disintegrated and then, after a preliminary gravitational separation, the final separation of water, oil and suspended solids is carried out in a number of centrifuges.

### The Shape of the Liquid Surface

Consider a centrifuge basket in which the axis of rotation is vertical (Fig. 13.5). Any element of liquid exerts a force $g$ per unit mass in a downward direction and $x\omega^2$ in a radial direction. If the element of liquid is situated at the free surface, the resultant force must act in a direction perpendicular to the surface. Taking the origin as the centre of the bottom of the basket, the slope of the liquid surface, at a point $(x, y)$ is given by:

$$\frac{\mathrm{d}y}{\mathrm{d}x} = \frac{x \text{ component of force}}{y \text{ component of force}}$$

$$= \frac{x\omega^2}{g} \qquad \ldots.(13.1)$$

Thus, on integration, $y = \dfrac{\omega^2}{2g} x^2 + \text{constant}$

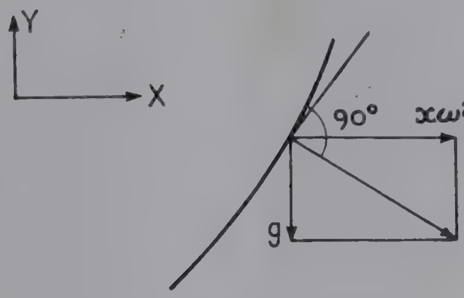


Fig. 13.5.
Element of surface of liquid

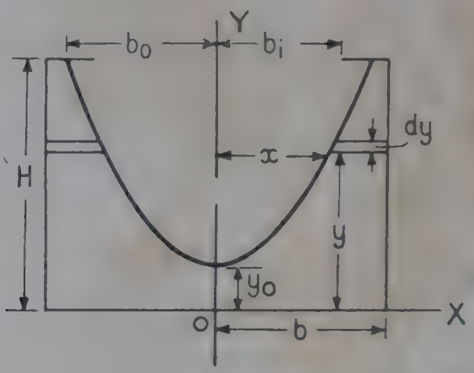


Fig. 13.6.
Volume of liquid in basket ($y_0$ positive)

When $x = 0$, $y = y_0$ (say), where $y_0$ is the depth of liquid at the centre of the basket. $y_0$ can be positive, indicating a real depth, or negative, indicating that there is no liquid at this point and that the surface of the liquid, if continuous, would be at a depth $y_0$ below the bottom of the basket at $x = 0$.

Thus the equation of a section of the surface through the centre of rotation is

$$y = \frac{\omega^2}{2g} x^2 + y_0 \qquad \ldots.(13.2)$$

This is the equation of a parabola and therefore the surface of the liquid is a paraboloid.

In general, $y_0$ will not be known but it can be evaluated in terms of the volume of liquid present in the basket. At high speeds of rotation, $\dfrac{\mathrm{d}y}{\mathrm{d}x}$ is high and the liquid surface is therefore almost vertical so that the paraboloid is not complete and $y_0$ is negative.

### Volume of Liquid in the Basket

It is necessary to consider two conditions separately; the first is where $y_0$ is positive and the second is where $y_0$ is negative.

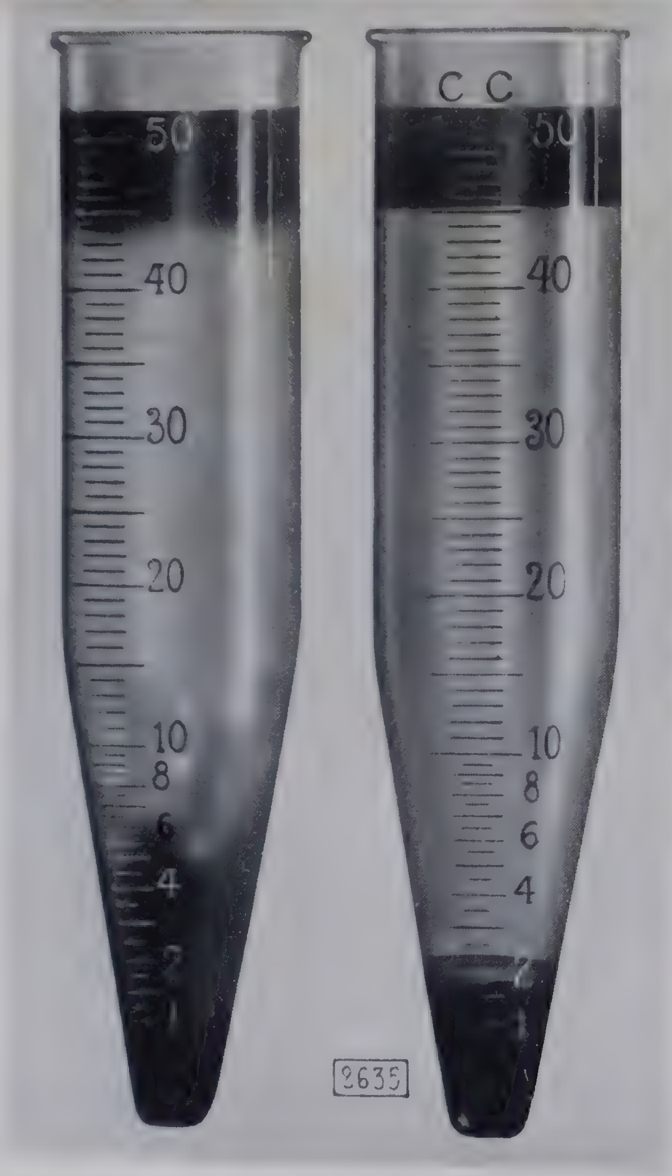

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Fig. 13.2. Results of gravity settling and centrifugal separation

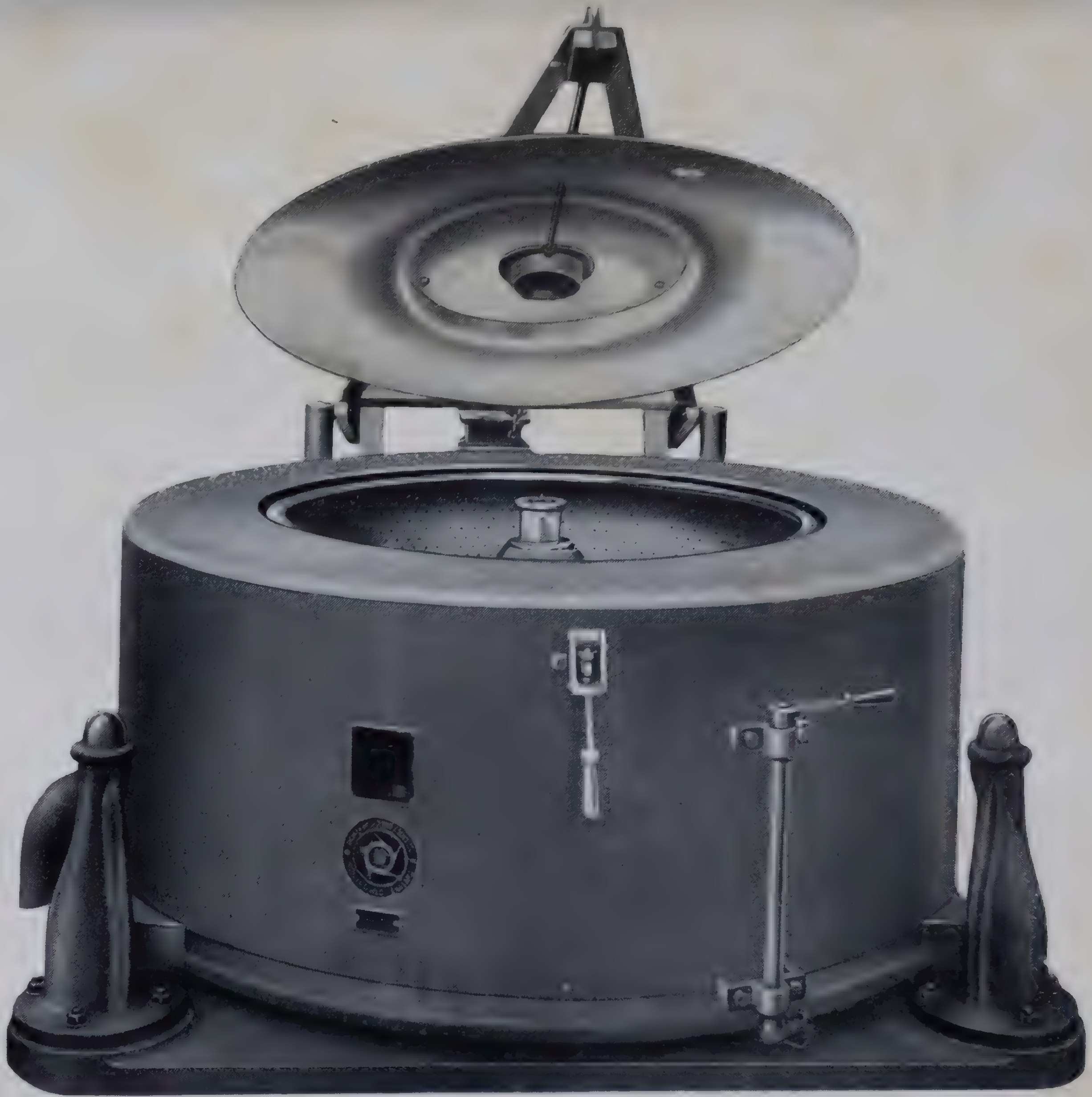

Fig. 13.3. Underdriven centrifuge

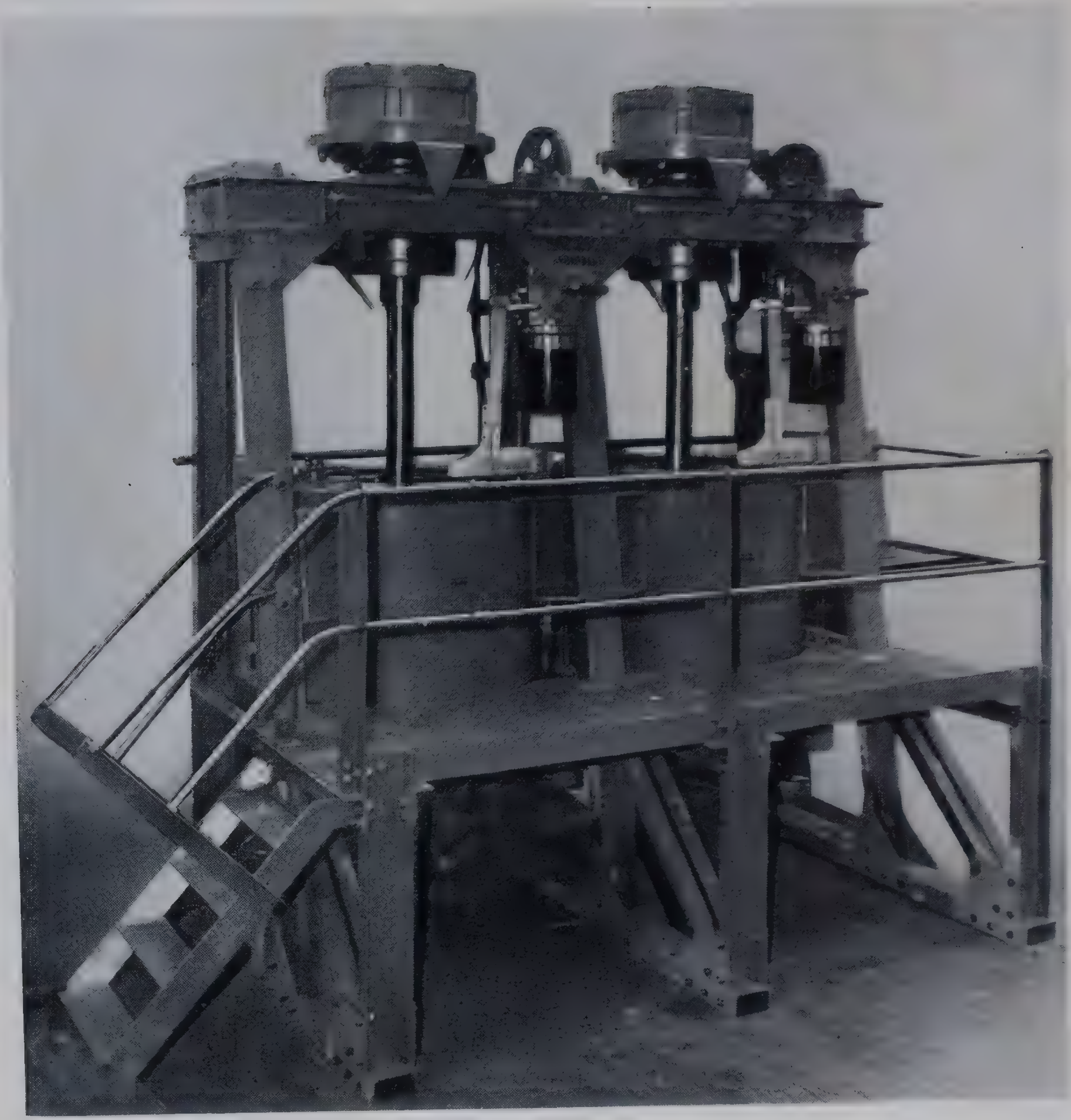

Fig. 13.4. Overdriven centrifuge

*$y_0$ positive* (Fig. 13.6)

The volume of liquid contained in a basket of radius $b$ and depth $H$,

$$\begin{aligned}
&= V' = \pi b^2 y_0 + \int_{y_0}^{H} \pi(b^2 - x^2)\,.\,\mathrm{d}y \\
&= \pi b^2 y_0 + \pi \int_{y_0}^{H} \left[b^2 - \frac{2g}{\omega^2}(y - y_0)\right].\,\mathrm{d}y \\
&= \pi b^2 y_0 + \pi(H - y_0)\left(b^2 + \frac{2gy_0}{\omega^2}\right) - \frac{\pi g}{\omega^2}(H^2 - y_0^2) \\
&= \pi\left\{b^2 y_0 + (H - y_0)\left[b^2 - \frac{g}{\omega^2}(H - y_0)\right]\right\} \\
&= \pi\left\{b^2 H - \frac{g}{\omega^2}(H - y_0)^2\right\} \qquad \ldots.(13.3)
\end{aligned}$$

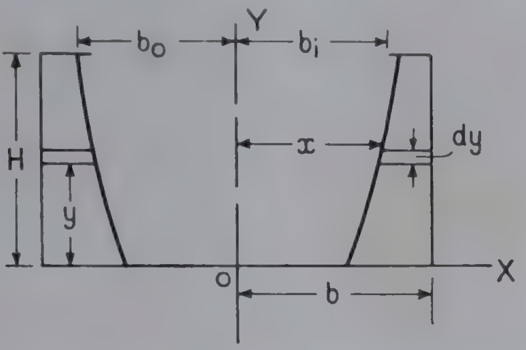

Fig. 13.7. Volume of liquid in basket ($y_0$ negative)

Thus $$y_0 = H - \sqrt{\frac{\omega^2}{g}\left(b^2 H - \frac{V'}{\pi}\right)} \qquad \ldots.(13.4)$$

Then substituting in equation 13.2

$$x^2 = \frac{2g}{\omega^2}\left(y - H + \sqrt{\frac{\omega^2}{g}\left(b^2 H - \frac{V'}{\pi}\right)}\right) \qquad \ldots.(13.5)$$

*$y_0$ negative* (Fig. 13.7)

$$\begin{aligned}
V' &= \int_0^H \pi(b^2 - x^2)\,\mathrm{d}y \\
&= \int_0^H \pi\left[b^2 - \frac{2g}{\omega^2}(y - y_0)\right]\mathrm{d}y \\
&= \pi\left(b^2 + \frac{2gy_0}{\omega^2}\right).\,H - \frac{\pi g}{\omega^2}.\,H^2 \\
&= \pi\left\{b^2 H - \frac{g}{\omega^2}(H^2 - 2y_0 H)\right\} \qquad \ldots.(13.6)
\end{aligned}$$

Thus $$y_0 = \tfrac{1}{2}\left\{H - \left(b^2 - \frac{V'}{\pi H}\right)\frac{\omega^2}{g}\right\} \qquad \ldots.(13.7)$$

and substituting in equation 13.2,

$$x^2 = \frac{2g}{\omega^2}\left\{y - \tfrac{1}{2}\left[H - \left(b^2 - \frac{V'}{\pi H}\right)\frac{\omega^2}{g}\right]\right\} \qquad \ldots.(13.8)$$

The limiting condition, when $y_0 = 0$, is obtained from either equation 13.3 or equation 13.6, giving

$$V' = \pi\left(b^2 H - \frac{g}{\omega^2} H^2\right). \qquad \ldots.(13.9)$$

Then if $V'$ exceeds this value, $y_0$ is positive: otherwise $y_0$ is negative.

The volume $V'$ of liquid in the basket can also be expressed in terms of the radius $b_0$ of the inner surface of the liquid at the top of the basket. The relation between $b_0$ and $y_0$ is obtained by substituting the co-ordinates, $x = b_0$, $y = H$, in equation 13.2 to give

$$y_0 = H - \frac{\omega^2}{2g} b_0^2 \qquad \ldots.(13.10)$$

The volume of liquid in the basket is then obtained by substituting this value of $y_0$ in equations 13.3 and 13.6, giving

$$V' = \pi\left(b^2 H - \frac{\omega^2 b_0^4}{4g}\right) \qquad \ldots.(13.11)$$

for $y_0$ positive and

$$V' = \pi\left(b^2 H + \frac{g}{\omega^2} H^2 - b_0^2 H\right) \qquad \ldots.(13.12)$$

for $y_0$ negative.

If the volume of liquid is small and the speed of rotation very low, the liquid level may not reach the top rim of the basket. Under normal conditions of operation, the centrifugal force will be so large compared with the gravitational force that the liquid surface will be almost vertical. Conditions in the basket are then independent of the direction of the axis of rotation.

### Fluid Pressure

The centrifugal pressure on the walls of the basket will now be calculated for a basket of radius $b$, on the assumption that the basket and contents are all rotating with the same angular velocity $\omega$.

Consider an element of liquid, depth $\mathrm{d}y$ and width $\mathrm{d}h$, subtending an angle $\mathrm{d}\theta$ at the centre. This element is situated at a height $y$ from the bottom of the basket, and $x$ is the radius of the inner surface of the liquid.

Centrifugal force exerted by element

$$= (x + h)\mathrm{d}\theta \,.\, \mathrm{d}h \,.\, \mathrm{d}y \,.\, \rho(x + h)\omega^2 \qquad \ldots.(13.13)$$

(where $\rho$ is the density of the liquid)

Then, if the centrifugal pressure in the liquid is $P_h$ at a radius $x + h$ and $P_h + \mathrm{d}P_h$ at a radius $x + h + \mathrm{d}h$, and if the pressure on the side boundaries is taken as a mean value $P_h + \frac{1}{2}\,dP_h$, as shown in Fig. 13.8, a force balance on the element in the radial direction gives:

$$P_h(x + h)\,d\theta\,.\,dy + 2(P_h + \tfrac{1}{2}\,dP_h)\,dh\,.\,dy\,\frac{d\theta}{2} - (P_h + dP_h)(x + h + dh)\,d\theta\,.\,dy$$

$$+ (x + h)\,d\theta\,.\,dh\,.\,dy\,.\,\rho\,(x + h)\omega^2 = 0$$

i.e. $$P_h(x + h) + P_h\,dh + \tfrac{1}{2}\,dP_h\,.\,dh - P_h(x + h) - P_h\,dh$$

$$- dP_h(x + h) - dP_h\,.\,dh + \rho(x + h)^2\omega^2\,dh = 0$$

Neglecting the terms which represent second order of small quantities,

$$dP_h = \rho\,.\,\omega^2(x + h)\,.\,dh \qquad \ldots.(13.14)$$

The centrifugal pressure $P_c$ on the walls of the basket is then given by integration of equation 13.14 between the limits of $h = 0$ and $h = b - x$.

i.e. $$P_c = \tfrac{1}{2}\rho\omega^2(b^2 - x^2) \qquad \ldots.(13.15)$$

The maximum possible value which the centrifugal pressure can attain occurs when $x = 0$ and is equal to

$$\tfrac{1}{2}\rho\omega^2 b^2. \qquad \ldots.(13.16)$$

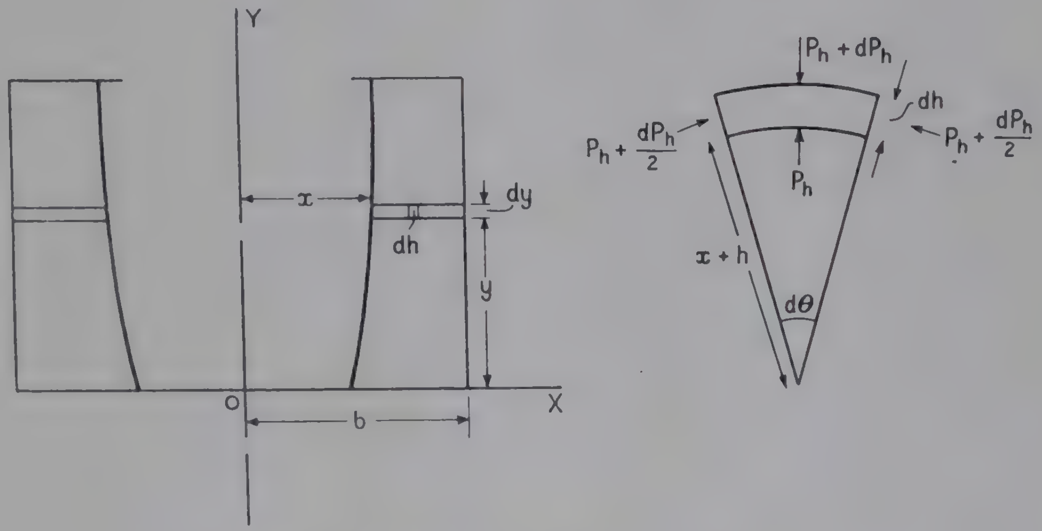

Fig. 13.8. Centrifugal pressure

The variation of the centrifugal pressure with height is readily obtained from equation 13.15 as follows:

$$P_c = \tfrac{1}{2}\rho\omega^2(b^2 - b_0^2) + \tfrac{1}{2}\rho\omega^2(b_0^2 - x^2)$$

From equation 13.2,

$$H - y = \frac{\omega^2}{2g}(b_0^2 - x^2) \qquad \ldots.(13.17)$$

$$P_c = \tfrac{1}{2}\rho\omega^2(b^2 - b_0^2) + (H - y)\rho g \qquad \ldots.(13.18)$$

Thus the pressure increases linearly with depth, and is equal to the centrifugal pressure at the top of the basket together with the hydrostatic pressure exerted by the fluid in the height $H - y$.

If the basket contains two immiscible liquids, the denser one will collect near the walls and the other will form an inner layer. Each will cause a centrifugal pressure to be exerted on the walls. If $x$ is the radius of the inner surface of the liquids, $x'$ is the radius of the interface between the two liquids, and $\rho$ and $\rho'$ are the densities of the heavy and light liquids respectively, the total centrifugal pressure is

$$\frac{\omega^2}{2}\left\{\rho'(x'^2 - x^2) + \rho(b^2 - x'^2)\right\}. \qquad \ldots.(13.19)$$

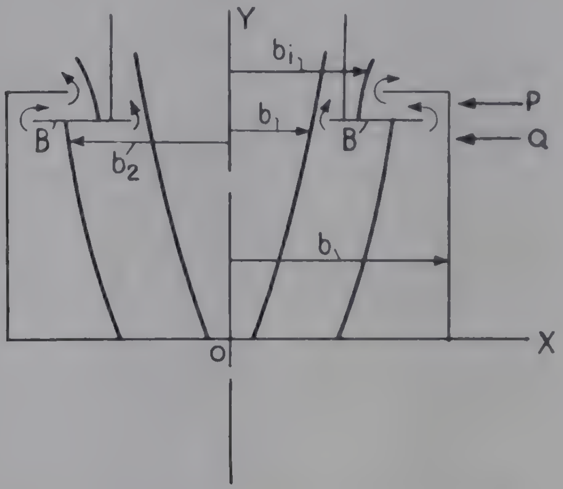

Fig. 13.9. Separation of two liquids

This expression can also be used to calculate the centrifugal pressure when the basket contains a liquid and a layer of solids deposited on the walls. In that case $\rho$ is the bulk density of the solid in the liquid and $\rho'$ is the density of the liquid. It is here assumed that the pressure is uniformly transmitted through the solid, as in a liquid.

## Separation of Two Immiscible Liquids

If a mixture of two liquids is to be continuously separated in a centrifuge; the position of the overflow weir must be carefully arranged in order to obtain a complete separation. Consider a basket of radius $b$ as shown in Fig. 13.9; at the top of the basket the radius of the inner surface of the liquid is $b_1$ and the radius of the interface between the liquids is $b_2$. The radius of the inner edge of the lip at the top of the basket is $b_i$. Then if the rate of passage of liquid through the centrifuge is not very high, $b_i$ will be approximately equal to the radius of the liquid surface. Now the centrifugal pressure on the walls of the basket will be the same at $P$ and $Q$ (Fig. 13.9) on each side of the baffle $B$ and thus from equation 13.19,

$$\frac{\omega^2}{2}\left\{\rho'(b_2^2 - b_1^2) + \rho(b^2 - b_2^2)\right\} = \frac{\omega^2}{2}\,\rho(b^2 - b_i^2)$$

$$\therefore \qquad \rho'(b_2^2 - b_1^2) = \rho(b_2^2 - b_i^2) \qquad \ldots.(13.20)$$

The required inner radius of the lip at the top of the basket will therefore be a function of the ratio of the densities of the two liquids, and of the radii $b_2$ and $b_1$ which are fixed by the flow rates of the two liquids.

For a given rate of feed to the centrifuge, the degree of separation obtained will depend on the thickness of the liquid layer formed at the walls of the basket and on the total depth of the basket, as this will control the time the mixture remains in the machine. A high degree of separation is therefore obtained with a long basket of small diameter, but the speed required is then very high. The introduction of conical discs in the bowl, as illustrated in Fig. 13.11, enables the liquid stream to be split into a large number of very thin layers in a bowl of much greater diameter. The separation of a mixture of water and dirt from a relatively low density oil takes place as shown in Fig. 13.10, with the dirt and water collecting close to the undersides of the discs and moving radially outwards, and with the oil moving inwards along the top sides. This bowl can be run at a very much lower speed and its size is very much smaller, as seen from Fig. 13.12. The separation of two liquids in a disc-type bowl is illustrated in the left-hand side of Fig. 13.11. Liquid enters through the distributor *AB*, passes through *C*, and is distributed between the discs *E* through the holes *D*. The denser liquid is taken off through *F* and *I* and the lighter liquid through *G*.

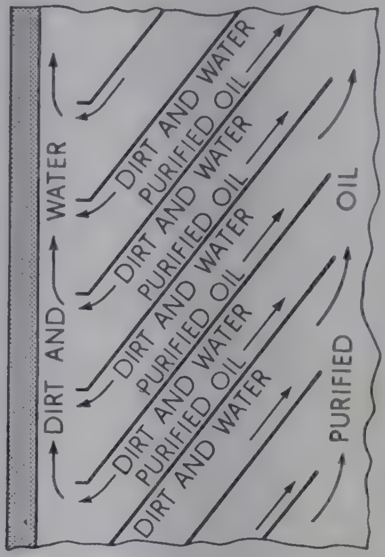

Fig. 13.10.
Separation of water and dirt from oil in disc bowl

## Separation of Solid from Liquid

The choice of method for separating solid particles from a liquid using a centrifuge depends on the resistance which the bed of deposited solids offers to the flow of liquid. With fine materials offering a high resistance to flow an imperforate basket is used, and the clarified liquid is removed either over a weir or through a stationary skimming tube. The suspension is continuously fed into the basket and the operation is continued until a suitable depth of solids has been deposited on the walls. The basket is then stopped and the solids are scraped off either by hand or by means of a scraper blade which slowly moves towards the surface of the basket as it is rotated by hand.

With a horizontally mounted basket, even conditions are obtained throughout the whole length, and the solids are more readily discharged because they can be allowed to fall directly into a chute. In the centrifuge shown in Figs. 13.13 and 13.14, the liquid is taken off through the skimming tube, and the cutter operates with the machine running at full speed so that a considerable saving of time and energy is achieved. The solids may be washed before the operation of the cutter. A similar machine with an inclined axis is shown in Fig. 13.15. A disc type of bowl is often used for the separation of fine solids from a liquid and its construction is shown in the right-hand side of Fig. 13.11. Here there is only one liquid outlet *K*, and the solids are retained in the space between the ends of the discs and the walls of the basket. The centrifuge can also be used as a classifier. The rate of feed and speed of rotation are so adjusted that the fine particles are carried away in the liquid overflow and the large ones are deposited on the walls of the basket. Solids and liquids are also separated in perforated baskets, as already mentioned. For relatively large crystals the inner surface of the basket is covered with a gauze which holds back the crystals but allows the liquid to pass. For the separation of small particles it is necessary to fix a filter cloth over the gauze.

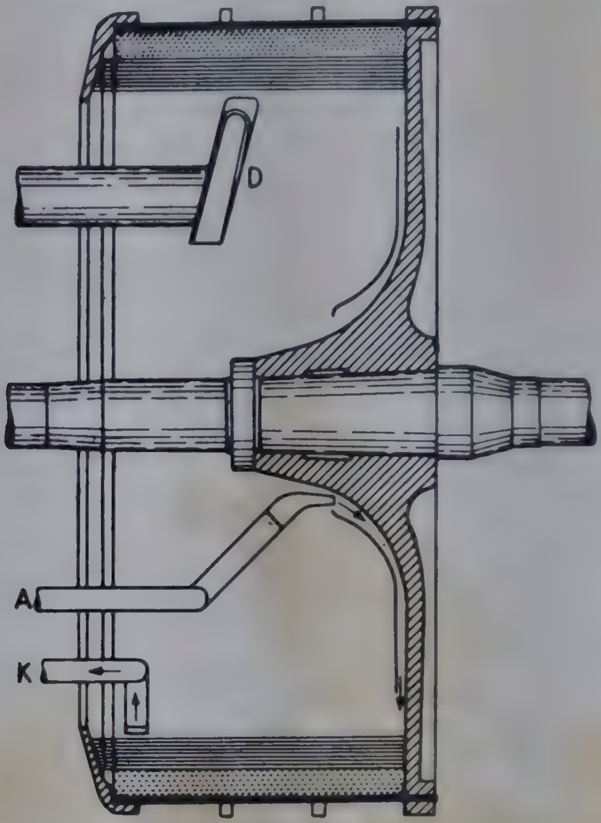

Fig. 13.13. Horizontally mounted basket with automatic discharge of solids
A—Feed D—Cutter K—Skimming tube

***Separation of Suspended Particles in a Centrifuge.*** The rate of separation in a centrifuge is considerably greater than that obtained with gravitational settling, but the settling velocity of the particles does not approach a terminal value because the accelerating force increases as the particles approach the walls of the basket. In a thin element of fluid at a distance $y$ from the bottom of the basket, the radius of the inner surface of the liquid is $x$. For a spherical particle of diameter $d$ under streamline conditions, the equation of motion of the particle in the radial direction is

$$\frac{\pi d^3}{6}(\rho_s - \rho) \, . \, (x + h)\,\omega^2 - 3\pi\mu d\frac{\mathrm{d}h}{\mathrm{d}t} - \frac{\pi d^3}{6}\rho_s\frac{\mathrm{d}^2h}{\mathrm{d}t^2} = 0$$

(It will be shown in Chapter 14, that the resistance force acting under these conditions is $3\pi\mu d\frac{\mathrm{d}h}{\mathrm{d}t}$). This equation can be solved only by graphical methods.

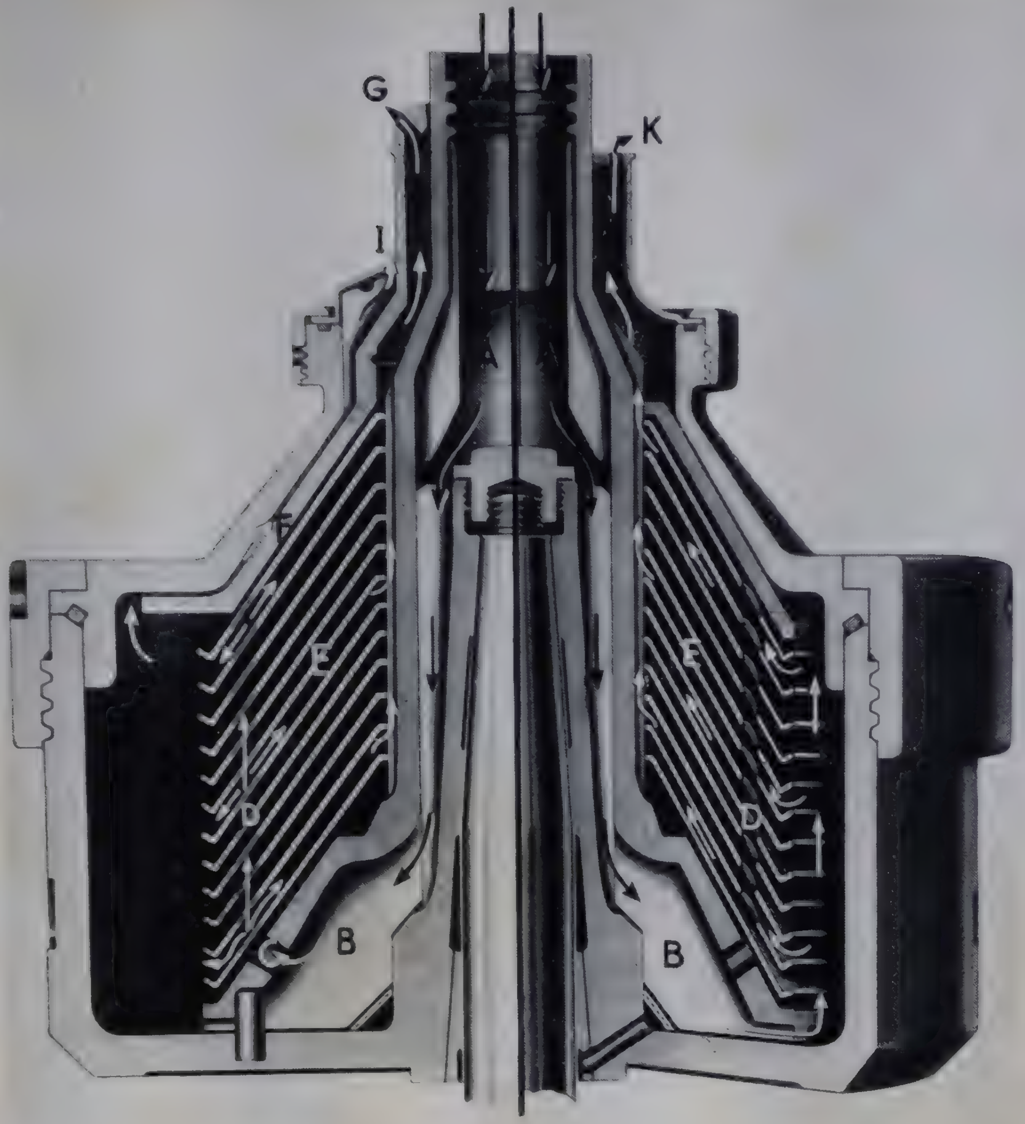

Fig. 13.11. Bowl with conical discs (left-hand side for separating liquids, right-hand side for separating solid from liquid)

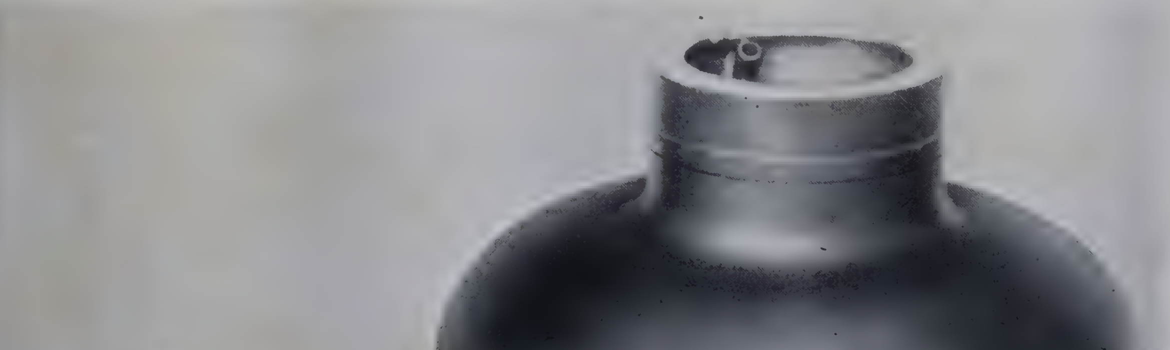

Fig. 13.12. Two bowls of equal capacity; with discs (left) and without discs (right)

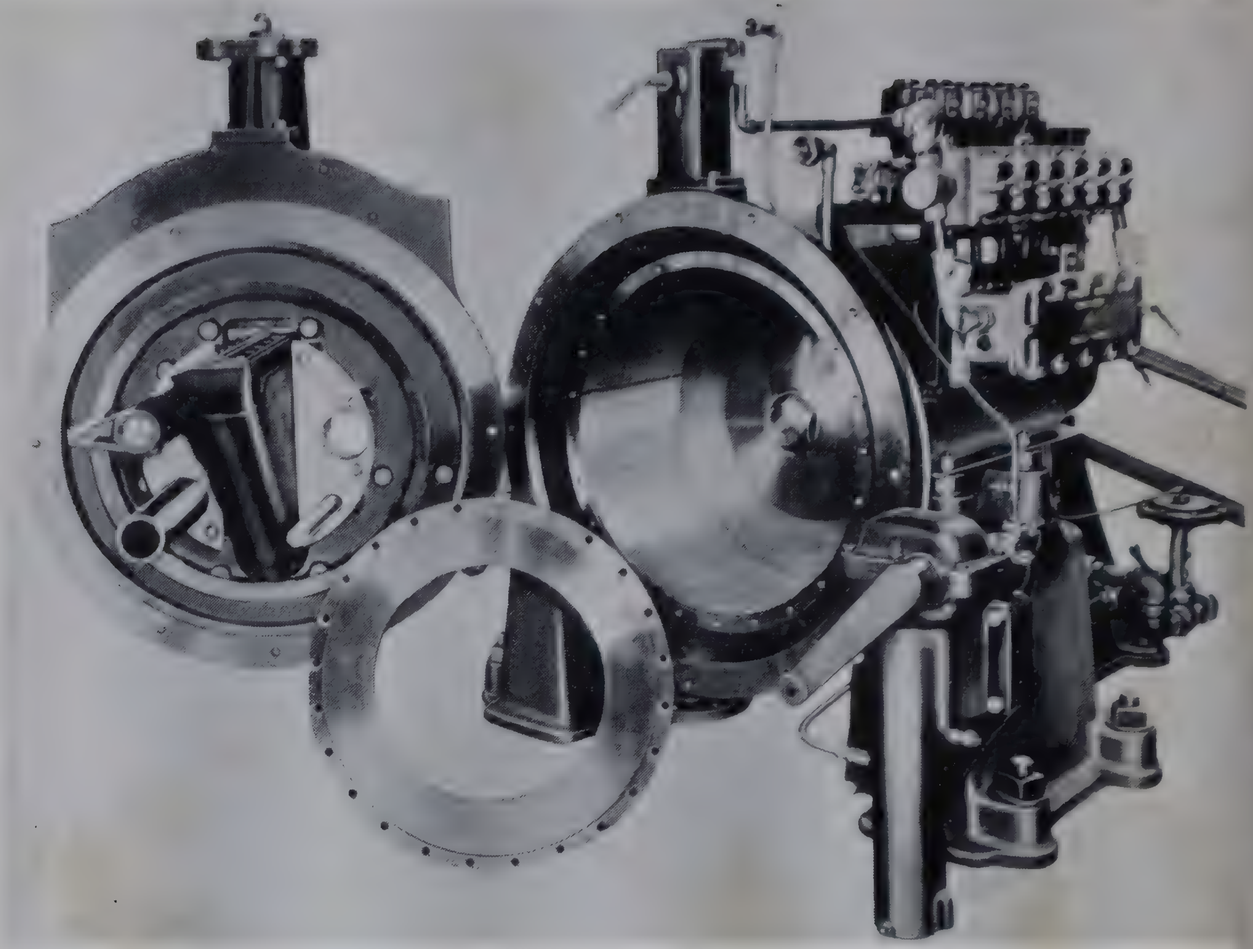

Fig. 13.14. Horizontal centrifuge with automatic discharge of solids

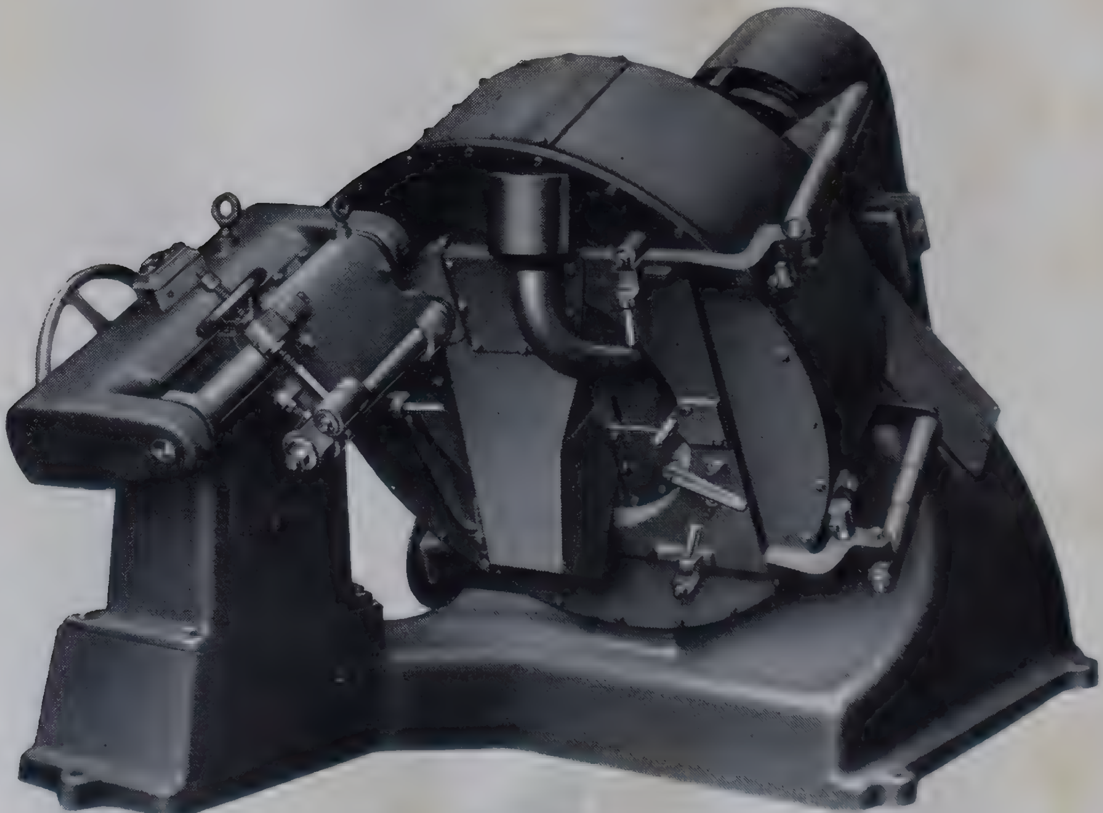

Fig. 13.15. Inclined centrifuge

However, if the acceleration term is neglected, the equation reduces to

$$\frac{\mathrm{d}h}{\mathrm{d}t} = \frac{d^2(\rho_s - \rho)\,.\,\omega^2}{18\mu}(x+h) \qquad \ldots.(13.21a)$$

i.e.

$$\mathrm{d}t = \frac{18\mu}{d^2\omega^2(\rho_s - \rho)}\,.\,\frac{\mathrm{d}(x+h)}{x+h}$$

The time $t$ taken for the particle to settle from a distance $h_1$ to a distance $h_2$ from the surface in a radial direction is then given by

$$t = \frac{18\mu}{d^2\omega^2(\rho_s - \rho)}\ln\frac{x+h_2}{x+h_1} \qquad \ldots.(13.21b)$$

If the motion of the fluid with respect to the particle is turbulent,

$$\frac{\pi d^3}{6}(\rho_s - \rho)\,.\,(x+h)\omega^2 - 0.22\left(\frac{\mathrm{d}h}{\mathrm{d}t}\right)^2\rho\,.\,\frac{\pi}{4}d^2 - \frac{\pi d^3}{6}\rho_s\,.\,\frac{\mathrm{d}^2h}{\mathrm{d}t^2} = 0$$

(It will be shown in Chapter 14 that the resistance force under these conditions is $0{\cdot}22\left(\frac{\mathrm{d}h}{\mathrm{d}t}\right)^2\rho\,.\,\frac{\pi}{4}d^2$).

Neglecting the acceleration of the particle,

$$\left(\frac{\mathrm{d}h}{\mathrm{d}t}\right)^2 = 3d\omega^2\left(\frac{\rho_s - \rho}{\rho}\right).\,(x+h)$$

$$\therefore \qquad \frac{\mathrm{d}(x+h)}{(x+h)^{\frac{1}{2}}} = a'\,\mathrm{d}t. \quad \text{where } a'^2 = 3d\omega^2\left(\frac{\rho_s - \rho}{\rho}\right)$$

and

$$t = \frac{2}{a'}\left\{(x+h_2)^{\frac{1}{2}} - (x+h_1)^{\frac{1}{2}}\right\} \qquad \ldots.(13.22)$$

These two equations can be used to determine approximately the time taken for a particle to travel from a radius of $x + h_1$ to $x + h_2$.

There is a maximum rate at which a suspension can be fed to the centrifuge in order to obtain complete separation, since an adequate time must be allowed for each particle to travel from the inner surface of the liquid to the walls of the basket before the liquid leaves the machine. If $x$ is constant, the maximum distance any particle must move is from $h = 0$ to $h + x = b$. The time taken can be calculated approximately from equations 13.21$b$ or 13.22 above, and since it must not be more than the time the particle remains in the basket, the maximum throughput of the machine can be calculated.

If the suspension is present as a thin film of thickness $h$ at the walls of a basket of radius $b$, we have from equation 13.21$a$ that

$$\frac{\mathrm{d}h}{\mathrm{d}t} = \frac{d^2(p_s - p)\,.\,\omega^2 b}{18\mu}$$

and the radial velocity of the particle can be taken as constant. The time any element of material remains in the basket is $Q/V'$, where $Q$ is the volumetric rate of feed to the centrifuge and $V'$ is the volume retained in the basket at any time. If the flow rate is so adjusted that a particle of diameter $d$ is just retained when it has to travel through the maximum distance $h$ before reaching the wall,

$$h = \frac{d^2(\rho_s - \rho)b\omega^2}{18\mu} \times \frac{V'}{Q}$$

i.e.

$$Q = \frac{d^2(\rho_s - \rho)b\omega^2 V'}{18\mu h}$$

$$= \frac{d^2(\rho_s - \rho)g}{18\mu} \times \frac{b\omega^2 V'}{gh} = u_0\Sigma \quad \text{(say)} \qquad \ldots.(13.23)$$

In equation 13.23, $u_0$ is the free settling velocity of a particle in the gravitational field (see Chapter 14), and $\Sigma$ is dependent solely on the dimensions of the centrifuge and the conditions of operation. $\Sigma$ is equal to the area of a gravity settling tank with the same clarifying capacity as the centrifuge, and therefore comparison of the values of $\Sigma$ for different machines provides a method of comparing their performances.

We have here considered a simple centrifuge with a cylindrical basket, but the same reasoning can be applied to a more complex shape, such as a basket with discs, if equivalent values of $b$ and $h$ are used. The value for a basket with discs will generally be considerably higher than that for the simple basket.

***Filtration in a Centrifuge.*** When filtration is carried out in a centrifuge, the driving force is the centrifugal pressure due to the liquid and, as indicated on page 453, this will not be affected by the presence of solid particles deposited on the walls. This force has to overcome the friction caused by the flow of liquid through the filter cake, the cloth, and the supporting gauze and perforations. The resistance of the filter cake will increase as solids are deposited but the other resistances will remain approximately constant throughout the process. Consider a filtration over a small length d$y$ at a distance $y$ from the closed end of a basket of radius $b$, and suppose that the suspension is introduced at such a rate that the inner radius of the liquid surface remains constant (Fig. 13.16). At some time $t$ after the commencement of filtration, a filter cake of thickness $l$ will have been built up and the radius of the interface between the cake and the suspension will be $b'$.

If d$P'$ is the pressure drop across a small thickness d$l$ of cake, the velocity of flow of the filtrate will be given by

$$u = \frac{\mathrm{d}P'}{r\mu \mathrm{d}l} \qquad \text{(from equation 12.2)}$$

where $r$ is the specific resistance of the filter cake and $\mu$ is the viscosity of the filtrate.

The filtrate will flow in an approximately radial direction and therefore the area available for flow will increase towards the walls of the basket. If $\mathrm{d}V$ is the volume of filtrate flowing through the small element of filter cake in time $t$,

$$u = \frac{1}{2\pi b'\mathrm{d}y} \cdot \frac{\mathrm{d}\,(\mathrm{d}V)}{\mathrm{d}t}$$

$$= \frac{1}{2\pi b'} \cdot \frac{\mathrm{d}^2 V}{\mathrm{d}y\,.\,\mathrm{d}t}$$

Thus

$$\frac{\mathrm{d}P'}{r\mu\mathrm{d}l} = \frac{1}{2\pi b'} \cdot \frac{\mathrm{d}^2 V}{\mathrm{d}y\,.\,\mathrm{d}t}$$

and

$$\mathrm{d}P' = \frac{r\mu\mathrm{d}l}{2\pi b'} \cdot \frac{\mathrm{d}^2 V}{\mathrm{d}y\,.\,\mathrm{d}t}$$

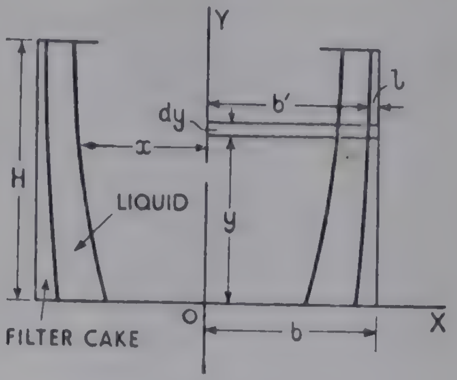

Fig. 13.16.
Filtration in a centrifuge

The total pressure drop through the cake at time $t$ is given by

$$\Delta P' = \frac{r\mu}{2\pi} \frac{\mathrm{d}^2 V}{\mathrm{d}y\,.\,\mathrm{d}t} \int_0^l \frac{\mathrm{d}l}{b'}$$

$$= \frac{r\mu}{2\pi} \frac{\mathrm{d}^2 V}{\mathrm{d}y\,.\,\mathrm{d}t} \int_b^{b'} \frac{\mathrm{d}(-b')}{b'}$$

$$= \frac{r\mu}{2\pi} \frac{\mathrm{d}^2 V}{\mathrm{d}y\,.\,\mathrm{d}t} \cdot \ln \frac{b}{b'} \qquad \ldots.(13.24)$$

If the resistance of the cloth is negligible and the centrifugal force is large compared with the gravitational force, $\Delta P'$ is equal to the centrifugal pressure, $\frac{1}{2}\rho\omega^2(b^2 - x^2)$ (from equation 13.15). We assume here that differences in density between the slurry and the filtrate are not large.

If the cloth is equivalent in resistance to a cake of thickness $L$, situated at the walls of the basket, the pressure drop $\Delta P''$ across the cloth is given by

$$\frac{\Delta P''}{r\mu L} = \frac{1}{2\pi b\,.\,\mathrm{d}y} \cdot \frac{\mathrm{d}\,(\mathrm{d}V)}{\mathrm{d}t}$$

i.e.

$$\Delta P'' = \frac{r\mu L}{2\pi b} \cdot \frac{\mathrm{d}^2 V}{\mathrm{d}y\,.\,\mathrm{d}t} \qquad \ldots.(13.25)$$

Thus the total pressure drop across the filter cake and the cloth,

$$= \Delta P' + \Delta P'' = \Delta P\,(\text{say}) = \tfrac{1}{2}\rho\omega^2(b^2 - x^2)\cdot \qquad \text{(from equation 13.16)}$$

and

$$\Delta P = \frac{r\mu}{2\pi} \cdot \frac{\mathrm{d}^2 V}{\mathrm{d}y\,.\,\mathrm{d}t} \left(\ln \frac{b}{b'} + \frac{L}{b}\right)\cdot \qquad \ldots.(13.26)$$

(from equations 13.24 and 13.25).
Before this equation can be integrated, it is necessary to establish the relation between $b'$ and $V$.

If $v$ is the bulk volume of incompressible cake deposited by the passage of unit volume of filtrate, the total volume of cake at time $t$ in the small element of basket depth $\mathrm{d}y$

$$= v\,.\,\mathrm{d}V = \pi(b^2 - b'^2)\,.\,\mathrm{d}y \qquad \ldots.(13.27)$$

$$\therefore \qquad v\,.\,\mathrm{d}(\mathrm{d}V) = -2\pi b'\,.\,\mathrm{d}b'\,.\,\mathrm{d}y$$

$$\frac{\mathrm{d}^2V}{\mathrm{d}y\,.\,\mathrm{d}t} = -\frac{2\pi b'}{v}\cdot\frac{\mathrm{d}b'}{\mathrm{d}t} \qquad \ldots.(13.28)$$

Substituting from equation 13.28 in equation 13.26,

$$\frac{r\mu}{2\pi} \times \left(-\frac{2\pi b'}{v}\cdot\frac{\mathrm{d}b'}{\mathrm{d}t}\right)\cdot\left(\ln\frac{b}{b'} + \frac{L}{b}\right) = \Delta P$$

$$\therefore \qquad -\frac{r\mu}{v}\cdot b'\frac{\mathrm{d}b'}{\mathrm{d}t}\left(\ln\frac{b}{b'} + \frac{L}{b}\right) = \Delta P$$

$$\therefore \qquad \left(\ln\frac{b'}{b} - \frac{L}{b}\right)\,.\,b'\,.\,\mathrm{d}b' = \frac{v\Delta P}{r\mu}\,.\,\mathrm{d}t \qquad \ldots.(13.29)$$

Integrating equation 13.29 between the limits $b' = b$ and $b' = b'$ as $t$ goes from 0 to $t$, ($\Delta P$ is constant because the inner radius of the liquid is maintained constant).

$$\left[\left(\ln\frac{b'}{b} - \frac{L}{b}\right)\,.\,\tfrac{1}{2}b'^2\right]_b^{b'} - \int_b^{b'}\frac{1}{b}\cdot\frac{b}{b'}\cdot\tfrac{1}{2}b'^2\mathrm{d}b' = \frac{\Delta Pvt}{r\mu}$$

$$\therefore \qquad \frac{L}{b}\cdot\tfrac{1}{2}b^2 + \left(\ln\frac{b'}{b} - \frac{L}{b}\right)\,.\,\tfrac{1}{2}b'^2 - \tfrac{1}{4}(b'^2 - b^2) = \frac{\Delta Pvt}{r\mu}$$

$$\therefore \qquad \tfrac{1}{4}(b^2 - b'^2) + \frac{L}{2b}(b^2 - b'^2) + \tfrac{1}{2}b'^2\ln\frac{b'}{b} = \frac{\Delta Pvt}{r\mu}$$

$$\therefore \qquad (b^2 - b'^2)\left(1 + 2\frac{L}{b}\right) + 2b'^2\ln\frac{b'}{b} = \frac{4\Delta Pvt}{r\mu}$$

$$= \frac{2vt\rho\omega^2}{r\mu}(b^2 - x^2)$$

$$\ldots.(13.30$$

From equation (13.30) the inner radius of the filter cake $b'$ at any time $t$ can b calculated, and hence the thickness can be obtained by subtracting $b'$ from $b$. th radius of the basket. The volume of filtrate obtained in the time $t$ is therefor given by equation 13.27,

$$v\,.\,\mathrm{d}V = \pi(b^2 - b'^2)\,.\,\mathrm{d}y$$

i.e.

$$V = \pi\int_0^H v^{-1}(b^2 - b'^2)\,.\,\mathrm{d}y \qquad \ldots.(13.3$$

Under normal operating conditions, $b'$ will be independent of $y$ and

$$V = \frac{\pi}{v}(b^2 - b'^2)H \qquad \ldots.(13.3$$

The cake will then be of the same thickness at all heights (except for any sagging which may occur).

HARUNI and STORROW(64) have carried out an extensive investigation of the flow of liquid through a cake formed in a centrifuge. They have concluded that, although the results of tests on a filtration plant and a centrifuge are often difficult to compare because of the effects of the compressibility of the cake, it is frequently possible to predict the flow rate in a centrifuge to within 20%. They have also shown that, when the thickness varies with height in the basket, the flow rate can be calculated on the assumption that the cake has a uniform thickness equal to the mean value; this gives a slightly high value in most cases.

**Example.** When an aqueous slurry is filtered in a plate and frame press, fitted with two 2-in. thick frames each 6 in. square, at 50 lb/sq in. gauge pressure the frames are filled in one hour. How long will it take to produce the same volume of filtrate as is obtained from a single cycle when using a centrifuge with a perforate basket, 12 in. diameter and 8 in. deep? The radius of the inner surface of the slurry is maintained constant at 3 in. and the speed of rotation is 4000 r.p.m.

Assume that the filter cake is incompressible and that the resistance of the cloth is equivalent to $\frac{1}{8}$ in. of cake in both cases.

**Solution**

*In filter press—*

$$V^2 + 2\frac{AL}{v}\cdot V = \frac{2\Delta P A^2 t}{r\mu v} \qquad (\because V = 0 \text{ when } t = 0) \qquad \text{(from equation 12.14a)}$$

Now

$$V = \frac{lA}{v} \qquad \text{(equation 12.6)}$$

$$\therefore \quad \frac{l^2A^2}{v^2} + \frac{2AL}{v}\cdot\frac{lA}{v} = \frac{2\Delta P A^2 t}{r\mu v}$$

$$\therefore \quad l^2 + 2Ll = \frac{2\Delta P v t}{r\mu}$$

*For one cycle—*

$$l = 1 \text{ in.} = \tfrac{1}{12} \text{ ft}, \quad L = \tfrac{1}{8} \text{ in.} = \tfrac{1}{96} \text{ ft}$$

$$\Delta P = 50 \text{ lb/sq in.} = 50 \times 144 \times 32{\cdot}2 = 232{,}000 \text{ poundals/sq ft}$$

$$t = 3600 \text{ sec.}$$

$$\therefore \quad \left(\frac{1}{12}\right)^2 \cdot \left(1 + \frac{1}{4}\right) = 2 \times 232{,}000 \times 3600 \times \frac{v}{r\mu}$$

$$\therefore \quad \frac{r\mu}{v} = \frac{464{,}000 \times 3600 \times 144}{1{\cdot}25}$$

$$= 19{\cdot}25 \times 10^{10}$$

*In the centrifuge—*

$$(b^2 - b'^2) . \left(2\frac{L}{b} + 1\right) + 2b'^2 . \ln\frac{b'}{b} = \frac{2vt\rho\omega^2}{r\mu}(b^2 - x^2) \qquad \ldots.(\text{Equation 13.30})$$

$$b = 6 \text{ in.} = 0{\cdot}5 \text{ ft}$$

$$H = 8 \text{ in.} = \frac{8}{12} \text{ ft}$$

$$\text{Volume of cake} = \frac{2 \times 2 \times 6^2}{1728} = \frac{1}{12} \text{ cu ft}$$

$$\therefore \quad \pi(b^2 - b'^2) \times \frac{8}{12} = \frac{1}{12}$$

$$\therefore \quad b'^2 = \frac{1}{4} - \frac{1}{8\pi} = 0{\cdot}2102 \text{ sq ft}$$

$$\therefore \quad b' = 0{\cdot}46 \text{ ft}$$

$$x = 3 \text{ in.} = 0{\cdot}25 \text{ ft}$$

$$\omega = \frac{4000}{60} \times 2\pi = 419 \text{ radians/sec.}$$

Time taken to produce same volume of filtrate or cake as in one cycle of the filter press is therefore given by—

$$\frac{1}{8\pi}\left(2 \times \frac{1/96}{0{\cdot}5} + 1\right) + 0{\cdot}2102 \ln\frac{0{\cdot}2102}{0{\cdot}25}$$

$$= \frac{2 \times t \times 62{\cdot}3 \times 419^2}{19{\cdot}25 \times 10^{10}}(0{\cdot}5^2 - 0{\cdot}25^2)$$

$$\therefore \quad 2{\cdot}13 \times 10^{-5}t = 0{\cdot}04144 - 0{\cdot}03645$$

$$\therefore \quad t = \frac{0{\cdot}0050}{2{\cdot}13 \times 10^{-5}}$$

$$= 235 \text{ seconds}$$

$$\approx \underline{\underline{4 \text{ minutes}}}$$

## Continuous Centrifuges

In one type (Fig. 13.17) of continuous centrifuge, the basket is either cylindrical or more commonly in the form of a truncated cone with its axis horizontal. The solids are continuously removed by means of a spiral scraper which rotates at a very slightly slower speed than the basket. The difference in speed is not sufficient to interfere with the separation but causes the removal of all the solids apart from a layer of thickness equal to the clearance between the scraper and the bowl. In many cases the basket and the scraper are driven by a single motor, but in the Dynocone separate drives are provided so that the differential speed can vary and the two can rotate without relative motion if the mechanism is overloaded. The suspension is fed to the centrifuge through a hollow shaft assisted by a screw mechanism if necessary, and separation is effected at the wide end of the basket. The solids are gradually moved along its surface and then come under the action of a spray of wash-liquid which also enters through the horizontal shaft. At the extreme end drying takes place and the solids are then discharged from the machine. The liquid is taken off, either through overflows if the basket is imperforate, or through the holes in a perforated basket.

In the push type of continuous centrifuge (Fig. 13.18), the feed is introduced through a conical funnel and the cake is formed in the space between the flange

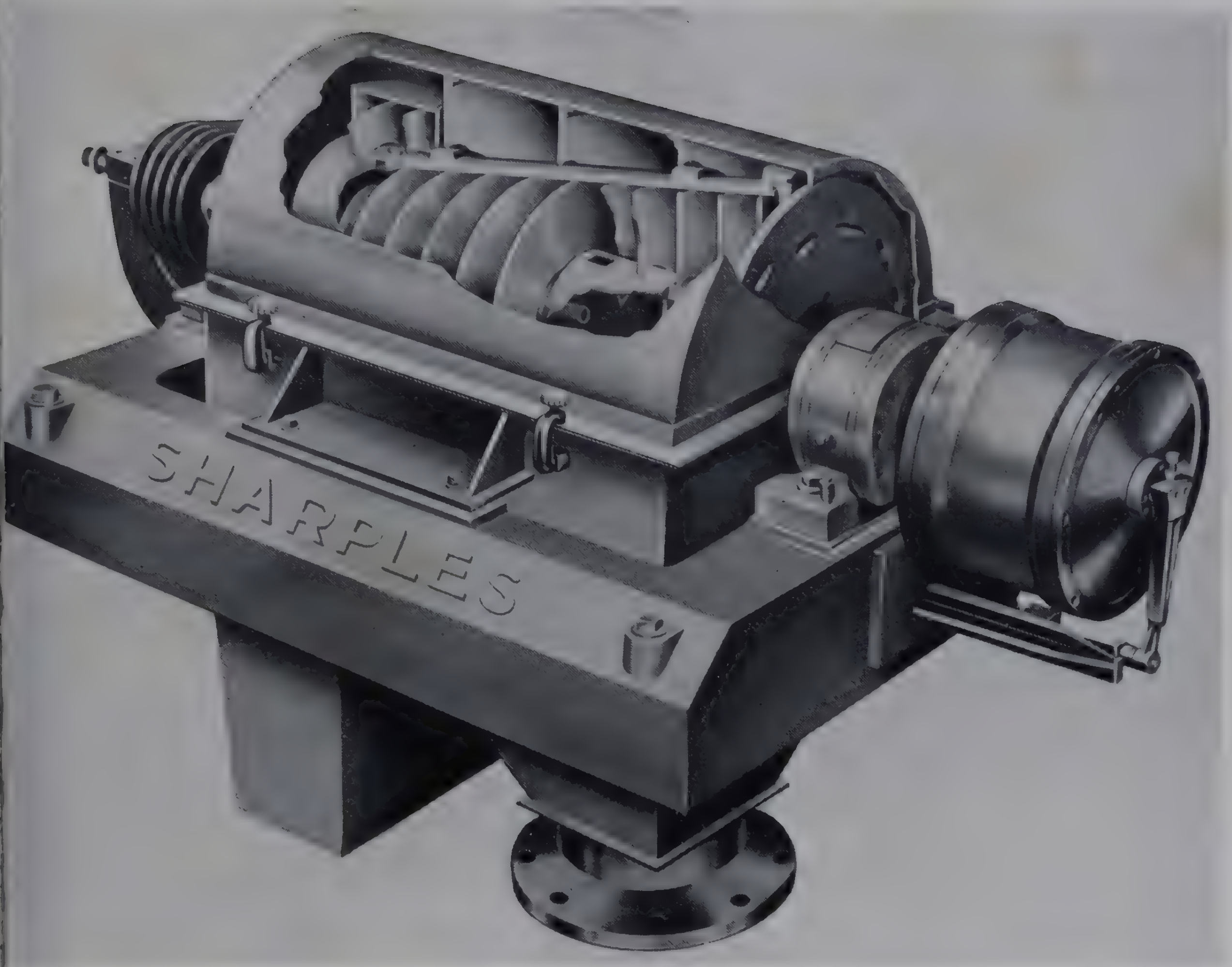

Fig. 13.17. Continuous conical centrifuge

and the end of the basket. The solids are intermittently moved along the surface of the basket by means of a reciprocating pusher. The pusher comes forward and returns immediately but waits until a further layer of solids has been built up before advancing again. In this machine the thickness of filter cake cannot exceed the distance between the surface of the basket and the flange of the funnel. The liquid either passes through the holes in the basket, or in the case of an imperforate basket is taken away through an overflow. The solids are washed by means of a spray, as shown.

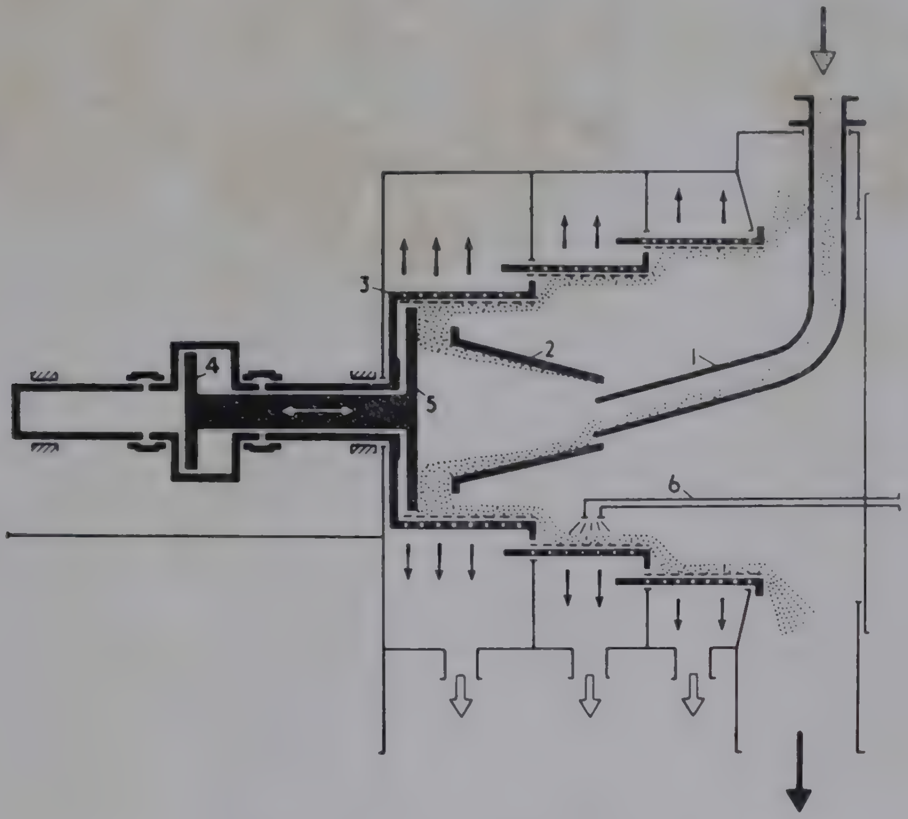

Fig. 13.18. Push type centrifuge

1. Inlet 2. Inlet funnel 3. Basket 4. Piston 5. Pusher disc 6. Washing spray

### The Supercentrifuge

It will be shown on page 471 that, for a given force acting on the material in the centrifuge, the stress in the walls of the basket is lowest when the radius is small. Thus when very high accelerating forces are required, it is desirable to use a basket of small diameter rotating at high speed. The supercentrifuge (Fig. 13.19) consists of a basket about 4 in. diameter and up to 3 ft 6 in. long; it usually incorporates baffles to bring the liquid rapidly up to speed. With speeds up to 60,000 r.p.m., accelerating forces up to 50,000 times the force of gravity are obtained.

For separation of colloidal particles and for breaking down emulsions the ultracentrifuge is used. It operates at speeds up to 100,000 r.p.m. and produces a force of as much as 500,000 times the force of gravity. The basket is usually driven by means of a small air turbine. The ultracentrifuge is often run either at low pressures or in an atmosphere of hydrogen in order to reduce frictional losses and a fivefold increase in the maximum speed can be attained by this means.

## Mechanical Design

Two aspects of the mechanical design of the centrifuge will be considered. The first is the thickness of the basket necessary for it to withstand the stresses set up during the most unfavourable conditions of operation. The other is the effect of uneven loading of the basket and the deflection in the driving shaft at various speeds of rotation. The treatment will be applicable both to perforated and imperforate baskets.

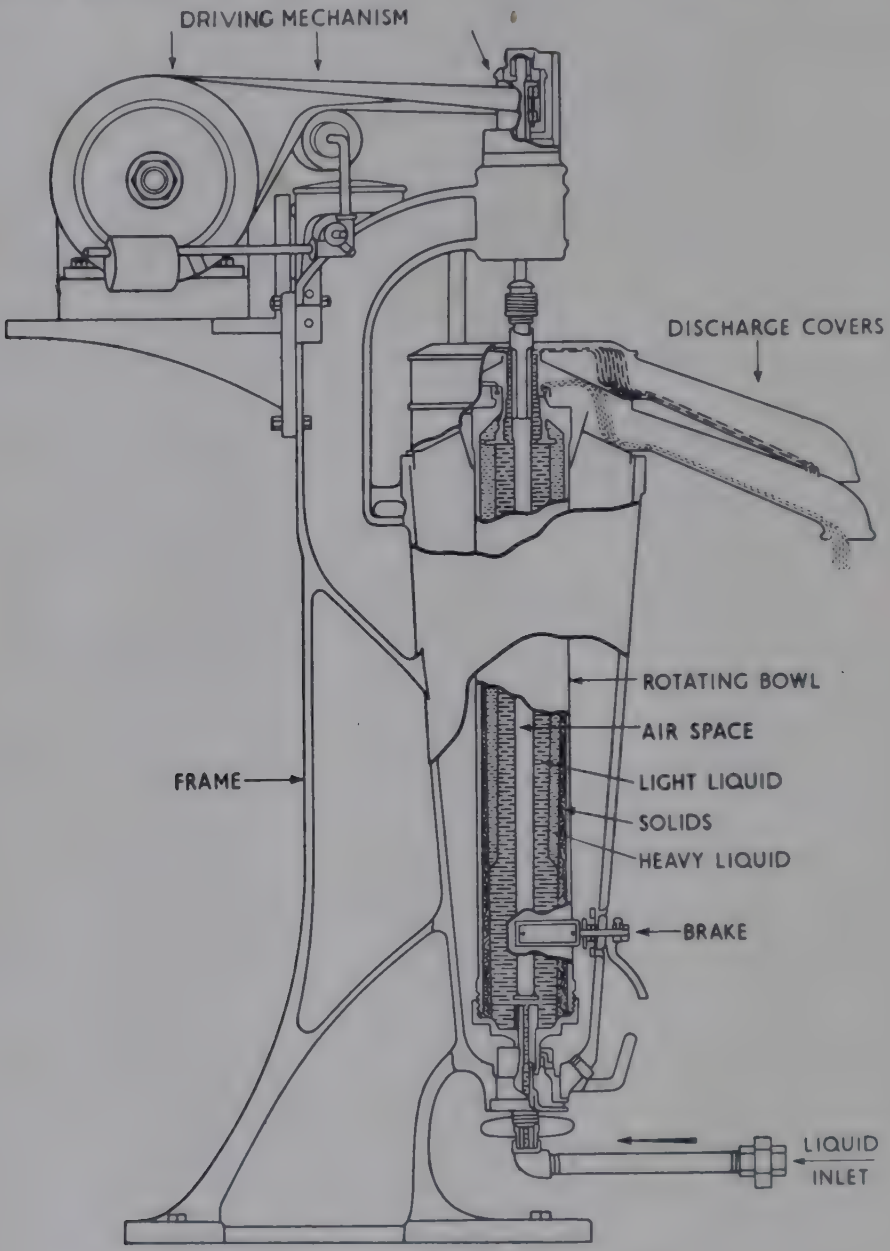
DRIVING MECHANISM
DISCHARGE COVERS
ROTATING BOWL
AIR SPACE
LIGHT LIQUID
SOLIDS
HEAVY LIQUID
FRAME
BRAKE
LIQUID INLET

Fig. 13.19. The supercentrifuge

***Wall Thickness.*** In a balanced centrifuge, the centrifugal force which acts on the walls of the basket is made up of three components, attributable to

(a) the material forming the basket,
(b) the solids deposited on the walls, and
(c) the liquid in the basket.

The loading will be a maximum at the bottom of the basket if the axis of rotation is vertical; and will decrease slightly towards the top. The centrifugal pressure due to a solid and a liquid has already been calculated (equation 13.19) and will be denoted by the symbol $P_c$.

In a basket of radius $b$ and thickness $\delta$, the centrifugal pressure resulting from the rotation of the empty basket is $\rho_m \delta b \omega^2$, where $\rho_m$ is the density of the material. The total centrifugal pressure is therefore $P_c + \rho_m \delta b \omega^2$. The total load on an element of the circumference of length $b \,.\, \mathrm{d}\theta$ and depth $\mathrm{d}y$ (Fig. 13.20)

$$\mathrm{d}F = b \,.\, \mathrm{d}\theta \,.\, \mathrm{d}y \,.\, (P_c + \rho_m \delta b \omega^2)$$

This load must be balanced by the radial component of the tension in the material. If $\mathrm{d}T$ is the tensile force acting on the element,

the radial component

$$= 2\mathrm{d}T \,.\, \sin(\tfrac{1}{2}\mathrm{d}\theta) = \mathrm{d}T \,.\, \mathrm{d}\theta$$

Thus $\quad \mathrm{d}T \,.\, \mathrm{d}\theta = b \,.\, \mathrm{d}\theta \,.\, \mathrm{d}y \,.\, (P_c + \rho_m \delta b \omega^2)$

The stress in the walls,

$$f = \frac{\mathrm{d}T}{\delta \mathrm{d}y} = \frac{b}{\delta}(P_c + \rho_m \delta b \omega^2) \quad \ldots .(13.33)$$

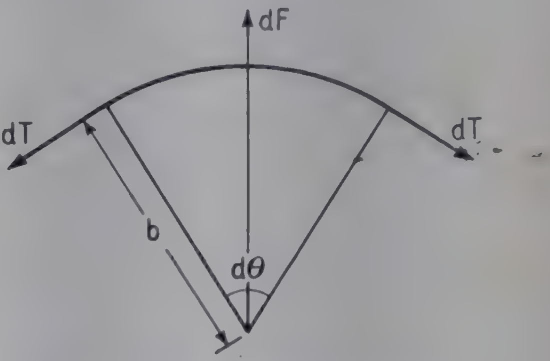

Fig. 13.20. Stress in basket

This stress must not exceed the limiting safe stress of the material. Thus the required thickness for a basket which is to operate under given conditions can be calculated. Alternatively the extreme conditions which can be used with a given basket can be determined.

If the basket is perforated, its mass will be less but the tensile force will be taken over a smaller area. In order to maintain the strength of the basket the perforations should be as small as possible and the basket is often hooped in order to compensate for the loss in strength caused by the perforations. The maximum safe stresses and details of operation are given in B.S.767.[68]

From equation 13.33, it is seen that for a given value of the centrifugal pressure $P_c$, the stress in the walls decreases as the radius of the basket decreases. Thus, when it is desired to produce very large accelerating forces, as in the ultracentrifuge, small diameter baskets are used at high speeds of rotation.

**Example.** What is the maximum safe speed of rotation of a phosphor bronze centrifuge basket, 12 in. diameter and $\frac{3}{16}$ in. thick, when it contains, at the walls, 3 in. of a liquid of specific gravity unity? Take the specific gravity of phosphor bronze as 8·9 and the safe working stress as 8000 lb/sq in.

**Solution.** From equation 13.15, the centrifugal pressure due to the liquid,

$$P_c = \tfrac{1}{2}\rho\omega^2(b^2 - x^2)$$

$$= \tfrac{1}{2} \times 62{\cdot}5 \times \omega^2(\tfrac{1}{4} - \tfrac{1}{16})$$

$$= 5{\cdot}9\omega^2 \text{ poundals/sq ft}$$

The stress in the walls of the basket is given by equation 13.33

$$
\begin{aligned}
f &= \frac{b}{\delta}(P_c + \rho_m \delta b \omega^2) \\
&= 32\,(5{\cdot}9\omega^2 + 8{\cdot}9 \times 62{\cdot}5 \times \tfrac{1}{64} \times \tfrac{1}{2} \times \omega^2) \\
&= 32\omega^2\,(5{\cdot}9 + 4{\cdot}3) \\
&= 327\omega^2 \text{ poundals/sq ft}
\end{aligned}
$$

The maximum safe stress $= 8000 \times 32{\cdot}2 \times 144 = 3{\cdot}71 \times 10^7$ poundals/sq ft

The maximum speed of rotation is therefore $\sqrt{\dfrac{3{\cdot}71 \times 10^7}{327}}$

$$
\begin{aligned}
&= 317 \text{ radians/sec} \\
&= \underline{\underline{3000 \text{ r.p.m.}}}
\end{aligned}
$$

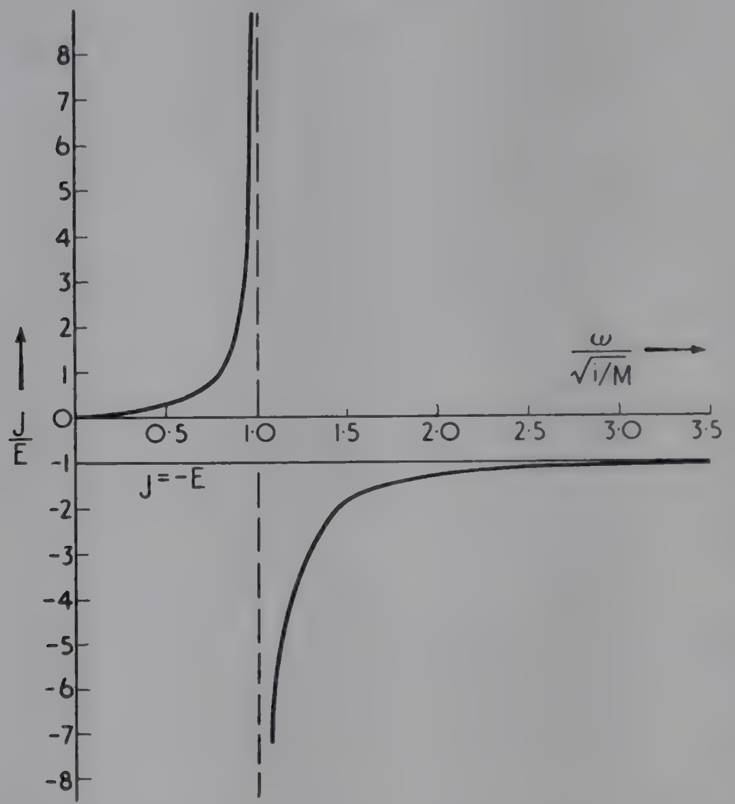

Fig. 13.21. Displacement of shaft of eccentrically mounted basket as a function of speed

***Critical Speed.*** If the loading on the centrifuge basket is uneven, the axis of rotation may not pass through the mass centre and a resultant force will be exerted on the shaft in a radial direction. Suppose that the mass centre is at a distance $E$ from the centre of rotation when the basket is stationary and that the shaft is deflected through a distance $j$ when the basket is rotating at an angular velocity $\omega$. Then, if $M$ is the mass of the basket and its contents, the resultant force$^{(14)}$ acting on the shaft will be approximately $M\omega^2(E + j)$. Then if the restoring force is approximately proportional to the deflection and equal to $i\,.\,j$ (say),

$$M\omega^2\,(E + j) = i\,.\,j$$

and therefore

$$
\begin{aligned}
j &= \frac{M\omega^2 E}{i - M\omega^2} \\
&= \frac{E}{i/M\omega^2 - 1} \qquad \ldots.(13.34)
\end{aligned}
$$

It is seen that the deflection $j$ increases as the speed of rotation increases until $\omega = \sqrt{\frac{i}{M}}$; the deflection then approaches infinity. This speed is known as the critical speed of the centrifuge; the machine would be expected to disintegrate when rotating at this speed. It does not in fact do so, though the deflection tends to become large, because $i$ does not remain constant at large displacements: in any case the machine would normally be brought through its critical speed very quickly. At speeds higher than the critical speed, the deflection of the shaft becomes negative, i.e. in the opposite direction to the initial eccentricity. At very high speeds the deflection becomes equal and opposite to the initial eccentricity and the basket tends to rotate about its mass centre. In Fig. 13.21, the calculated deflection of the shaft $j$ is plotted against the angular velocity $\omega$.

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## LIST OF SYMBOLS USED IN CHAPTERS 11, 12, 13

| | | |
|---|---|---|
| $A$ | Cross-sectional area of bed or filtration area | $L^2$ |
| $a$ | Surface of solids per unit volume of bed | $L^{-1}$ |
| $a'$ | $\sqrt{3d\omega^2\left(\frac{\rho_s-\rho}{\rho}\right)}$ | $L^{\frac{1}{2}}T^{-1}$ |
| $B_1$ | Coefficient | $L^{-6}T$ |
| $B_2$ | Coefficient | $L^{-3}T$ |
| $b$ | Radius of basket of centrifuge | $L$ |
| $b'$ | Radius of inner surface of filter cake at height $y$ | $L$ |
| $b_l$ | Inner radius of lip of basket | $L$ |
| $b_0$ | Radius of inner surface of liquid at top of basket | $L$ |
| $b_1$ | Radius of inner surface of inner liquid | $L$ |
| $b_2$ | Radius of interface between two liquids | $L$ |
| $c$ | Mass of solids deposited by passage of unit volume of filtrate | $ML^{-3}$ |
| $D$ | Characteristic dimension of particle | $L$ |

| | | |
|---|---|---|
| $d$ | Equivalent spherical diameter of particles | L |
| $d_1$ | Diameter of capillary | L |
| $d_n$ | Nominal size of Raschig ring (*centimetres*) | L |
| $d_p$ | Linear dimension of particle | L |
| $d'_m$ | One quarter of hydraulic mean diameter of pore spaces | L |
| $E$ | Eccentricity of basket | L |
| $e$ | Voidage of bed or filter cake | — |
| $e_z$ | Voidage at distance $z$ from surface | — |
| $e'$ | Voidage near walls of container | — |
| $F$ | Force on circumference of basket | $MLT^{-2}$ |
| $f$ | Stress in walls of basket | $ML^{-1}T^{-2}$ |
| $f_1, f_2$ | Correction factors | — |
| $G$ | Mass rate of flow of gas | $MT^{-1}$ |
| $g$ | Acceleration due to gravity | $LT^{-2}$ |
| $H$ | Depth of basket | L |
| $H'$ | Depth of liquid at walls of basket | L |
| $h$ | Radial distance from inner surface of liquid | L |
| $I$ | A coefficient | $(ML^{-1}T^{-2})^{-n'}$ |
| $i$ | Ratio of restoring force to deflection | $MT^{-2}$ |
| $J$ | Mass fraction of solids in slurry | — |
| $j$ | Displacement of shaft of centrifuge | L |
| $K$ | A coefficient | $M^{-1}L^3T$ |
| $K'$ | A constant | — |
| $K''$ | KOZENY's constant | — |
| $k'$ | Constant for given shape of particle | — |
| $k''$ | Constant for a given depth of bed | — |
| $L$ | Thickness of filter cake with same resistance as cloth | L |
| | *or* mass rate of flow of liquid | $MT^{-1}$ |
| $l$ | Thickness of filter cake or bed, or side of cube | L |
| $l'$ | Length of capillary tube | L |
| $M$ | Mass of basket and contents | M |
| $m$ | Mass of particle | M |
| $N$ | Number of particles in bed | — |
| $n, n'$ | Indices | — |
| $P_1$ | Pressure at downstream face of cake | $ML^{-1}T^{-2}$ |
| $P_2$ | Pressure at upstream face of cake | $ML^{-1}T^{-2}$ |
| $P_c$ | Centrifugal pressure | $ML^{-1}T^{-2}$ |
| $P_h$ | Pressure in liquid in centrifuge at distance $h$ from surface | $ML^{-1}T^{-2}$ |
| $P_z$ | Pressure at distance $z$ from surface | $ML^{-1}T^{-2}$ |
| $\Delta P$ | Total difference in pressure | $ML^{-1}T^{-2}$ |
| $\Delta P'$ | Pressure difference across cake | $ML^{-1}T^{-2}$ |
| $\Delta P''$ | Pressure difference across cloth | $ML^{-1}T^{-2}$ |
| $\Delta P_d$ | Pressure difference across dry column | $ML^{-1}T^{-2}$ |
| $\Delta P_w$ | Pressure difference across wet-drained column | $ML^{-1}T^{-2}$ |
| $Q$ | Volumetric rate of feed to centrifuge | $L^3T^{-1}$ |
| $R'$ | Component of drag force, per unit area of particle surface | $ML^{-1}T^{-2}$ |
| $R''$ | Quantity proportional to $R'$ | $ML^{-1}T^{-2}$ |
| $r$ | Specific resistance of filter cake | $L^{-2}$ |
| $r''$ | Function of $\bar{r}_z$, independent of velocity | $M^{-1}L^{1-n}T^n$ |
| $r_0$ | Function of $\bar{r}_z$, independent of velocity and pressure difference | $M^{-1}L^{1-n}T^n$ |
| $r_z$ | Specific resistance at distance $z$ from surface | $L^{-2}$ |
| $\bar{r}$ | Specific resistance based on unit mass of cake | $M^{-1}L$ |
| $\bar{r}', \bar{r}''$ | Functions of $\bar{r}$ independent of $\Delta P$ | $M^{-(n'+1)}L^{n'+1}T^{2n'}$ |
| $\bar{r}'''$ | Function of $\bar{r}$ independent of $\Delta P$ | $M^{-1}L$ |
| $\bar{r}_z$ | Value of $\bar{r}$ at distance $z$ from surface | $M^{-1}L$ |

| | | |
|---|---|---|
| $S$ | Specific surface | $L^{-1}$ |
| $S_c$ | Surface of containing vessel per unit volume of particle | $L^{-1}$ |
| $s$ | Saturation of bed | — |
| $s_e$ | Effective saturation | — |
| $s_f$ | Fraction of voids in which no fluid is flowing | — |
| $s_r$ | Residual saturation | — |
| $T$ | Tensile force in basket | $MLT^{-2}$ |
| $t$ | Time | $T$ |
| $t'$ | Time of dismantling filter press | $T$ |
| $t_1$ | Time at beginning of operation | $T$ |
| $U$ | Resistance of filter medium | $L^{-1}$ |
| $u$ | Mean velocity of flow calculated over whole area | $LT^{-1}$ |
| $u'$ | Velocity of gas produced by same pressure difference in dry bed | $LT^{-1}$ |
| $u_1$ | Average velocity in tube or channel | $LT^{-1}$ |
| $V$ | Volume of liquid flowing in time $t$ | $L^3$ |
| $V'$ | Volume of liquid in basket | $L^3$ |
| $V_1$ | Volume of liquid passing in time $t_1$ | $L^3$ |
| $v$ | Volume of cake deposited by unit volume of filtrate | — |
| $W$ | Overall volumetric rate of filtration | $L^3T^{-1}$ |
| $w$ | Hydrostatic head | $L$ |
| $x$ | Radius of inner surface of liquid at height $y$ | $L$ |
| $x'$ | Radius of interface between two liquids | $L$ |
| $y$ | Distance from bottom of basket | $L$ |
| $y_0$ | Depth of liquid at centre of basket | $L$ |
| $Z$ | Constant in correlation for flooding in liquid-liquid system | — |
| $z$ | Distance from surface of filter cake | $L$ |
| $\alpha$, $\alpha'$ | Coefficients | $ML^{-2}T^{-1}$, $ML^{-(n+1)}T^{n-2}$ |
| $\delta$ | Thickness of basket | $L$ |
| $\mu$ | Viscosity of fluid | $ML^{-1}T^{-1}$ |
| $\mu_w$ | Viscosity of water at 15°C | $ML^{-1}T^{-1}$ |
| $\rho$ | Density of fluid | $ML^{-3}$ |
| $\rho'$ | Density of lighter liquid | $ML^{-3}$ |
| $\rho_m$ | Density of material of basket | $ML^{-3}$ |
| $\rho_s$ | Density of solids | $ML^{-3}$ |
| $\Delta\rho$ | $\lvert(\rho_c - \rho_d)\rvert$ | $ML^{-3}$ |
| $\Sigma$ | Capacity factor for centrifuge | $L^2$ |
| $\sigma$ | Interfacial tension | $MT^{-2}$ |
| $\sigma_T$ | Interfacial tension, air-water at 15°C | $MT^{-2}$ |
| $\phi$ | ROSE's friction factor | — |
| $\phi'$ | CHILTON and COLBURN's friction factor | — |
| $\psi$ | Sphericity of particle | — |
| $\theta$ | Angle subtended by portion of circumference at centre | — |
| $\omega$ | Angular velocity | $T^{-1}$ |
| *Ca.* | Capillary number | — |
| *Re.'* | Modified Reynolds Number used by CARMAN | — |
| *Re."* | Modified Reynolds Number used by CHILTON and COLBURN, and ROSE | — |

Suffixes $G$, $L$, $c$, $d$ refer to gas, liquid, continuous phase and disperse phase.

## Section F

# *Systems involving Relative Motion between a Fluid and Particles*

## CHAPTER 14

# *Motion of Particles in a Fluid*

A NUMBER of processes for the separation of particles of various sizes and shapes depend on the variation in the behaviour of the particles when they are subjected to the action of a moving fluid. Further, many of the methods for the determination of the sizes of particles in the sub-sieve ranges involve relative motion between the particles and a fluid.

The flow problems so far considered have been essentially unidirectional, with the fluid flowing along a pipe or channel, and the effect of an obstruction has been discussed only in so far as it causes an alteration in the forward velocity of the fluid. In the present chapter, we shall be concerned with the force exerted on a body as a result of the flow of fluid past it and, as in general the fluid will be diverted all round it, we are faced with the more complex problem of three dimensional flow. We shall now consider the flow of fluid relative to an infinitely long cylinder, a spherical particle and a non-spherical particle. The problem is essentially the same whether the fluid or the particle is moving.

### FLOW PAST A CYLINDER

The flow of fluid past an infinitely long cylinder, in a direction perpendicular to its axis, will be considered in the first instance because this involves only two directional flow, there being no flow parallel to the axis. For a non-viscous fluid flowing past a cylinder, as shown in Fig. 14.1, the velocity and direction of flow will vary round the circumference. Thus at *A* and *D* the fluid is brought to rest and at *B* and *C* the velocity is a maximum. Since the fluid is non-viscous, there will be no drag, and an infinite velocity gradient will exist at the surface of the cylinder. If the fluid is incompressible and the cylinder is small, the sum of the kinetic energy and the pressure energy is constant at all points on the surface. The kinetic energy is a maximum at *B* and *C* and zero at *A* and *D*, so that the pressure falls from *A* to *B* and from *A* to *C* and rises again from *B* to *D* and from *C* to *D*, the pressure at *A* and *D* being the same. There will therefore be no net force exerted by the fluid on the cylinder. It is found that, whereas the predicted pressure variation for a non-viscous fluid agrees well with the results obtained with a viscous fluid over the front face, very considerable differences occur at the rear face.

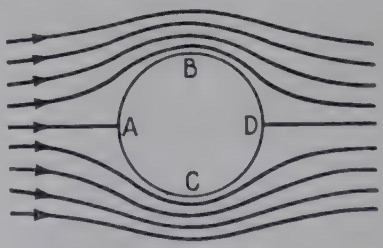


Fig. 14.1.
Flow round a cylinder

It has already been shown in Chapter 9 that, when a viscous fluid flows over a surface, the fluid is retarded in the boundary layer which is formed near the surface and that the boundary layer increases in thickness with increase in distance from the leading edge. If the pressure is falling in the direction of flow, the retardation of the fluid will be less and the boundary layer will be thinner in consequence. On the other hand, if the pressure is rising, there will be a greater retardation and the thickness of the boundary layer will increase more rapidly. The force acting on the fluid at some point in the boundary layer may then be sufficient to bring it to rest or to cause flow in the reverse direction with the result that an eddy current is set up. A region of reverse flow then exists near the surface where the boundary layer has separated (Fig. 14.2). The velocity rises from zero at the surface to a maximum negative value and falls again to zero; it then increases in the positive direction until it reaches the main stream velocity at the edge of the boundary layer, as seen in Fig. 14.2. At *PQ* the velocity in the *X*-direction is zero and the direction of flow in the eddies must be in the *Y*-direction.

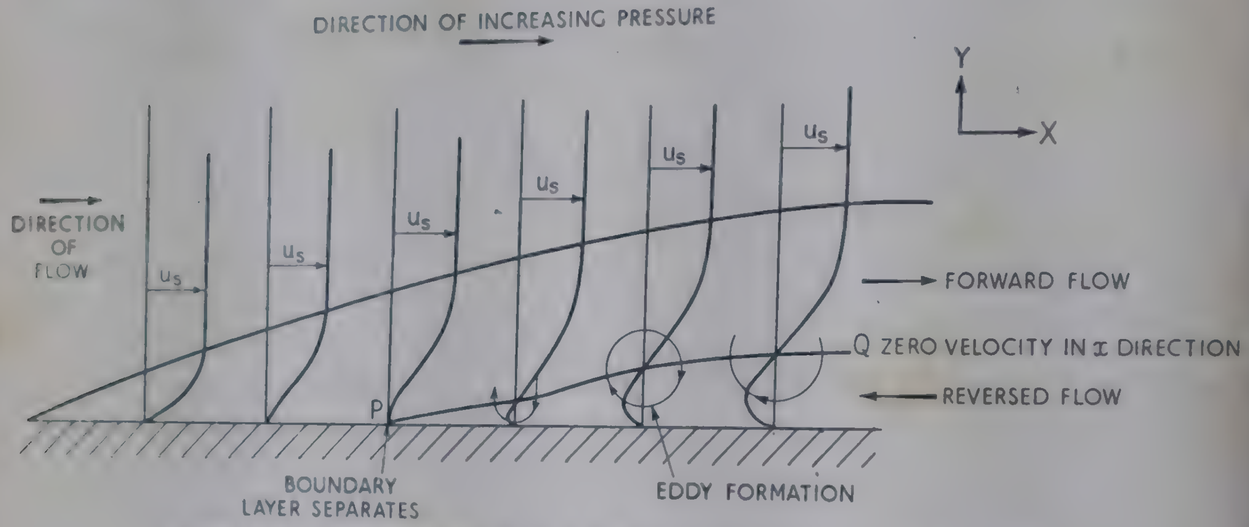
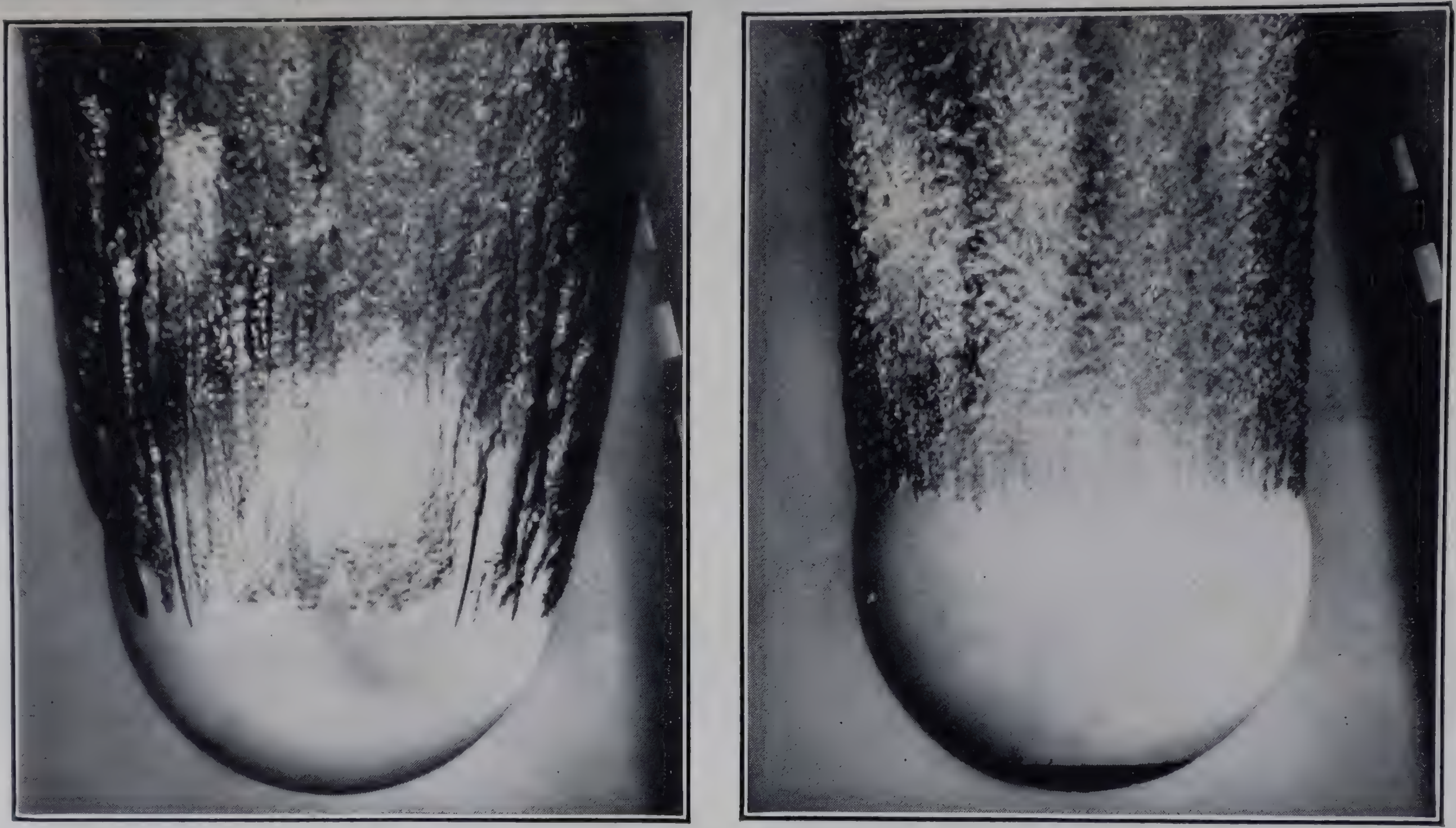


Fig. 14.2. Flow of fluid over a surface against a pressure gradient

For the flow of a viscous fluid past the cylinder, the pressure decreases from *A* to *B* and from *A* to *C* so that the boundary layer is thin and the flow is similar to that obtained with a non-viscous fluid: from *B* to *D* and from *C* to *D* the pressure is rising and therefore the boundary layer rapidly thickens with the result that it tends to separate from the surface. If separation occurs, eddies are formed in the wake of the cylinder and energy is thereby dissipated and an additional force, known as form drag, is set up. Thus, on the forward surface of the cylinder, the pressure distribution is similar to that obtained with the ideal fluid of zero viscosity; but on the rear surface, the boundary layer is thickening rapidly and pressure variations are very different in the two cases.

The total force on a body immersed in a moving fluid is made up of two components, the viscous drag or skin friction, and the form drag. At low rates of flow no separation of the boundary layer takes place and the whole of the drag results from skin friction. As the velocity is increased, separation of the boundary layer occurs and the skin friction forms a gradually decreasing proportion of the total drag. However, if the velocity of the fluid is very high, or if turbulence is artificially induced, the flow within the boundary layer will



(*a*) (*b*)

Fig. 14.3. Effect of roughening front face of a sphere

(*a*) 8½ in. diameter ball entering water at 25°C.
(*b*) As above, except for 4 in. diameter patch of sand on nose.

change from streamline to turbulent before separation takes place. Since the rate of transfer of momentum through a fluid in turbulent motion is much greater than that in a fluid flowing under streamline conditions, separation is less likely to occur, because the fast moving fluid outside the boundary layer is able to keep the fluid within the boundary layer moving in the forward direction. If separation does occur, it will take place nearer to $D$ in Fig. 14.1, and the resulting eddies will be smaller and the total drag will be reduced.

Turbulence may arise either from an increased fluid velocity or from artificial roughening of the forward face of the immersed body. PRANDTL roughened the forward face of a sphere by fixing a hoop to it with the result that the drag was considerably reduced. Further experiments have been carried out in which sand particles have been stuck to the front face (see Fig. 14.3). The tendency for separation and hence the magnitude of the form drag are also dependent on the shape of the body.

Conditions of flow relative to a spherical particle are similar to those relative to the cylinder except that the flow pattern is three directional. The method of calculating the force on a sphere is discussed in the following section.

## THE DRAG FORCE ON A SPHERICAL PARTICLE

The only practically important case for which the drag on an immersed body has been calculated from purely theoretical considerations is that of a sphere moving at a low velocity in an infinite extent of continuous fluid. In 1851 STOKES(5) obtained the formula,

$$F = 3\pi \mu d u \qquad \ldots.(14.1)$$

where $F$ is the drag force,

$\mu$ is the viscosity of the fluid,
$d$ is the diameter of the sphere, and
$u$ is the velocity of the fluid relative to the particle.

The experimental data for the drag force exerted on a spherical particle by a moving fluid are conveniently expressed over a wide range of velocities in a manner similar to that used for flow in a pipe, by plotting a dimensionless group $\frac{R'}{\rho u^2}$ against a modified Reynolds Group $Re.'$, in which the linear dimension is the diameter of the sphere, and the velocity is the relative velocity between the fluid and the particle. $Re.'$ is then equal to $\frac{ud\rho}{\mu}$, where $\rho$ is the density of the fluid. $R'$ is the force exerted on unit projected area of particle in a plane at right angles to its direction of motion: this area is thus equal to that of a circle of the same diameter as the sphere.

Values of $\frac{R'}{\rho u^2}$ are given as a function of $Re.'$ in Table 14.1 and $\log \frac{R'}{\rho u^2}$ is plotted against $\log Re.'$ in Figs. 14.4 and 14.5. The curve can be divided into four regions as follows.

TABLE 14.1. $R'/\rho u^2$, $R'/\rho u^2 \, . \, Re.'^2$ and $R'/\rho u^2 \, . \, Re.'^{-1}$ *as functions of* $Re$.

| $Re.'$ | $R'/\rho u^2$ | $R'/\rho u^2 \, . \, Re.'^2$ | $R'/\rho u^2 \, . \, Re.'^{-1}$ |
|---|---|---|---|
| $10^{-3}$ | 12,000 | | |
| $2 \times 10^{-3}$ | 6,000 | | |
| $5 \times 10^{-3}$ | 2,400 | | |
| $10^{-2}$ | 1,200 | $1\cdot20 \times 10^{-1}$ | $1\cdot20 \times 10^{5}$ |
| $2 \times 10^{-2}$ | 600 | $2\cdot40 \times 10^{-1}$ | $3\cdot00 \times 10^{4}$ |
| $5 \times 10^{-2}$ | 240 | $6\cdot00 \times 10^{-1}$ | $4\cdot80 \times 10^{4}$ |
| $10^{-1}$ | 124 | 1·24 | $1\cdot24 \times 10^{3}$ |
| $2 \times 10^{-1}$ | 63 | 2·52 | $3\cdot15 \times 10^{2}$ |
| $5 \times 10^{-1}$ | 26·3 | 6·4 | $5\cdot26 \times 10$ |
| $10^{0}$ | 13·8 | $1\cdot38 \times 10$ | $1\cdot38 \times 10$ |
| $2 \times 10^{0}$ | 7·45 | $2\cdot98 \times 10$ | 3·73 |
| $5 \times 10^{0}$ | 3·49 | $8\cdot73 \times 10$ | $7\cdot00 \times 10^{-1}$ |
| 10 | 2·08 | $2\cdot08 \times 10^{2}$ | $2\cdot10 \times 10^{-1}$ |
| $2 \times 10$ | 1·30 | $5\cdot20 \times 10^{2}$ | $6\cdot50 \times 10^{-2}$ |
| $5 \times 10$ | 0·768 | $1\cdot92 \times 10^{3}$ | $1\cdot54 \times 10^{-2}$ |
| $10^{2}$ | 0·547 | $5\cdot47 \times 10^{3}$ | $5\cdot50 \times 10^{-3}$ |
| $2 \times 10^{2}$ | 0·404 | $1\cdot62 \times 10^{4}$ | $2\cdot02 \times 10^{-3}$ |
| $5 \times 10^{2}$ | 0·283 | $7\cdot08 \times 10^{4}$ | $5\cdot70 \times 10^{-4}$ |
| $10^{3}$ | 0·221 | $2\cdot21 \times 10^{5}$ | $2\cdot21 \times 10^{-4}$ |
| $2 \times 10^{3}$ | 0·22 | $8\cdot8 \times 10^{5}$ | $1\cdot1 \times 10^{-4}$ |
| $5 \times 10^{3}$ | 0·22 | $5\cdot5 \times 10^{6}$ | $4\cdot4 \times 10^{-5}$ |
| $10^{4}$ | 0·22 | $2\cdot2 \times 10^{7}$ | $2\cdot2 \times 10^{-5}$ |
| $2 \times 10^{4}$ | 0·22 | | |
| $5 \times 10^{4}$ | 0·22 | | |
| $10^{5}$ | 0·22 | | |
| $2 \times 10^{5}$ | 0·05 | | |
| $5 \times 10^{5}$ | 0·05 | | |
| $10^{6}$ | 0·05 | | |
| $2 \times 10^{6}$ | 0·05 | | |
| $5 \times 10^{6}$ | 0·05 | | |
| $10^{7}$ | 0·05 | | |

When $0\cdot2 < Re.' < 1000$, $\quad R'/\rho u^2 = 12 \, Re.'^{-1}(1 + 0\cdot15 \, Re.'^{0\cdot687})$

When $Re.' \nless 0\cdot2$, $\quad R'/\rho u^2 = 12 \, Re.'^{-1}$

$$R'/\rho u^2 \, . \, Re.'^2 = 12 \, Re.'$$

$$R'/\rho u^2 \, . \, Re.'^{-1} = 12 \, Re.'^{-2}$$

When $Re.' > 1000$ (but $< 10^5$), $\quad R'/\rho u^2 = 0\cdot22$

$$R'/\rho u^2 \, . \, Re.'^2 = 0\cdot22 \, Re.'^2$$

$$R'/\rho u^2 \, . \, Re.'^{-1} = 0\cdot22 \, Re.'^{-1}$$

When $Re.' >_{ca} 10^5$, $\quad R'/\rho u^2 = 0\cdot05.$

*Region (a)* $(10^{-4} < Re.' < 0\cdot2)$. The equation of the curve approximates to

$$\frac{R'}{\rho u^2} = 12 \, Re.'^{-1} \qquad \ldots .(14.2)$$

i.e. on the logarithmic scale it is a straight line of slope $-1$. It will be shown later that equations 14.1 and 14.2 are consistent.

*Region (b)* $(0\cdot2 < Re.' < 500–1000)$

Various attempts have been made to derive theoretical expressions which can be used at higher values of $Re'$. For instance, GOLDSTEIN(15) has given an infinite series for the drag force on a spherical particle. This can be used for values of *Re.'* up to about 2, but at higher values, the series either converges very slowly or diverges. His relation is

$$\frac{R'}{\rho u^2} = \frac{12}{Re.'}\left\{1 + \frac{3}{16}Re.' - \frac{19}{1280}Re.'^2 + \frac{71}{20480}Re.'^3 - \frac{30179}{34406400}Re.'^4 + \frac{122519}{560742400}Re.'^5 - \dots\right\} \qquad \dots.(14.3)$$

From this expression it is seen that equation 14.2 is in error by nearly 4 per cent at a value of *Re.'* of 0·2.

The slope of the curve gradually changes from $-1$ to 0 as *Re.'* changes from 0·2 to 500 or 1000. SCHILLER and NAUMANN(16) have given the following empirical equation to cover this range.

$$\frac{R'}{\rho u^2} = 12\,Re.'^{-1}(1 + 0{\cdot}15\,Re.'^{0{\cdot}687}) \qquad \dots.(14.4)$$

Equation 14.4 is reliable for values of *Re·'* up to about 800. Some typical values of $R'/\rho u^2$ calculated from this formula are as follows:

$Re.' = 0{\cdot}2$ $\quad R'/\rho u^2 = 12\,Re.'^{-1} \times 1{\cdot}05$
$Re.' = 2{\cdot}0$ $\quad R'/\rho u^2 = 12\,Re.'^{-1} \times 1{\cdot}24$
$Re.' = 500$ $\quad R'/\rho u^2 = 0{\cdot}28$
$Re.' = 800$ $\quad R'/\rho u^2 = 0{\cdot}24$

Various other approximate formulae have been given for the drag force for this region. ALLEN(6) has considered the curve as having a mean slope of $-\frac{1}{2}$ and suggested that the data can be represented by the formula $R'/\rho u^2 = \text{constant} \times Re.'^{-\frac{1}{2}}$.

*Region* (*c*). The value of $R'/\rho u^2$ remains approximately constant at 0·22 for values of *Re.'* between about 500–1000 and $2 \times 10^5$. The data can therefore be represented by the equation,

$$\frac{R'}{\rho u^2} = 0{\cdot}22. \qquad \dots.(14.5)$$

*Region* (*d*). When *Re.'* exceeds about $2 \times 10^5$, the flow in the boundary layer changes from streamline to turbulent and the separation takes place nearer to the rear of the sphere and the drag force is decreased considerably and

$$\frac{R'}{\rho u^2} = 0{\cdot}05 \qquad \dots.(14.6)$$

The curve shown in Figs. 14.4 and 14.5 is really continuous and its division into four regions is only a convenient method by which a series of equations can be assigned to it. In region (*a*) it is assumed that skin friction is solely responsible for the drag, and in regions (*c*) and (*d*) that only form drag is acting. In fact, over the whole range of Reynolds Numbers both skin friction and form drag are present to some extent but the two are of comparable magnitude only in region (*b*). The exact division between regions (*a*) and (*b*), and between regions (*b*) and (*c*), depends on the limits at which form drag and skin friction respectively are assumed to become significant. Thus the upper limit for equation 14.2 is set by the accuracy required. As this is a simple equation to use, it is often applied for Reynolds Numbers up to about 2, but the calculated values of $\frac{R'}{\rho u^2}$ are then far less accurate over this extended range,

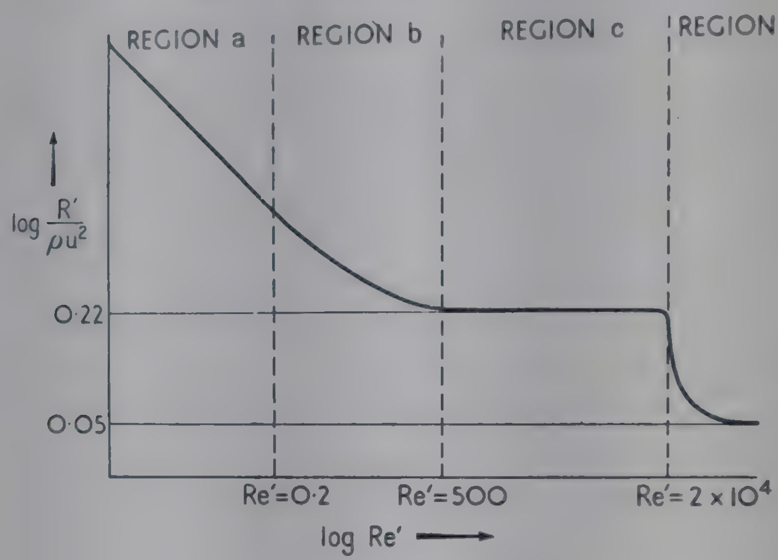


Fig. 14.4. Relation between $\frac{R'}{\rho u^2}$ and *Re.′* for a sphere

Although $\frac{R'}{\rho u^2}$ is shown exactly constant at 0·22 in region (*c*), it is often reported that the value gradually rises as the Reynolds Number increases, but the accuracy with which the relations can be applied seldom warrants a refinement of this sort.

**Total Force on the Particle**

In region (*a*),

$$R' = 12\rho u^2 \cdot \frac{\mu}{ud\rho} = \frac{12u\mu}{d} \qquad \ldots.(14.7)$$

The projected area of the particle $= \frac{1}{4}\pi d^2$

Thus the total force on the particle $F$

$$= \frac{12u\mu}{d} \times \frac{1}{4}\pi d^2 = 3\pi\mu du \qquad \ldots.(14.8)$$

This is the expression originally obtained by STOKES(5) (equation 14.1).

In region (*b*), from equation 14.4 we have the approximate relation,

$$R' = \frac{12u\mu}{d}(1 + 0{\cdot}15\, Re.'^{0\cdot687}) \qquad \ldots.(14.9)$$

and ∴

$$F = 3\pi\mu du\,(1 + 0{\cdot}15\, Re.'^{0\cdot687}) \qquad \ldots.(14.10)$$

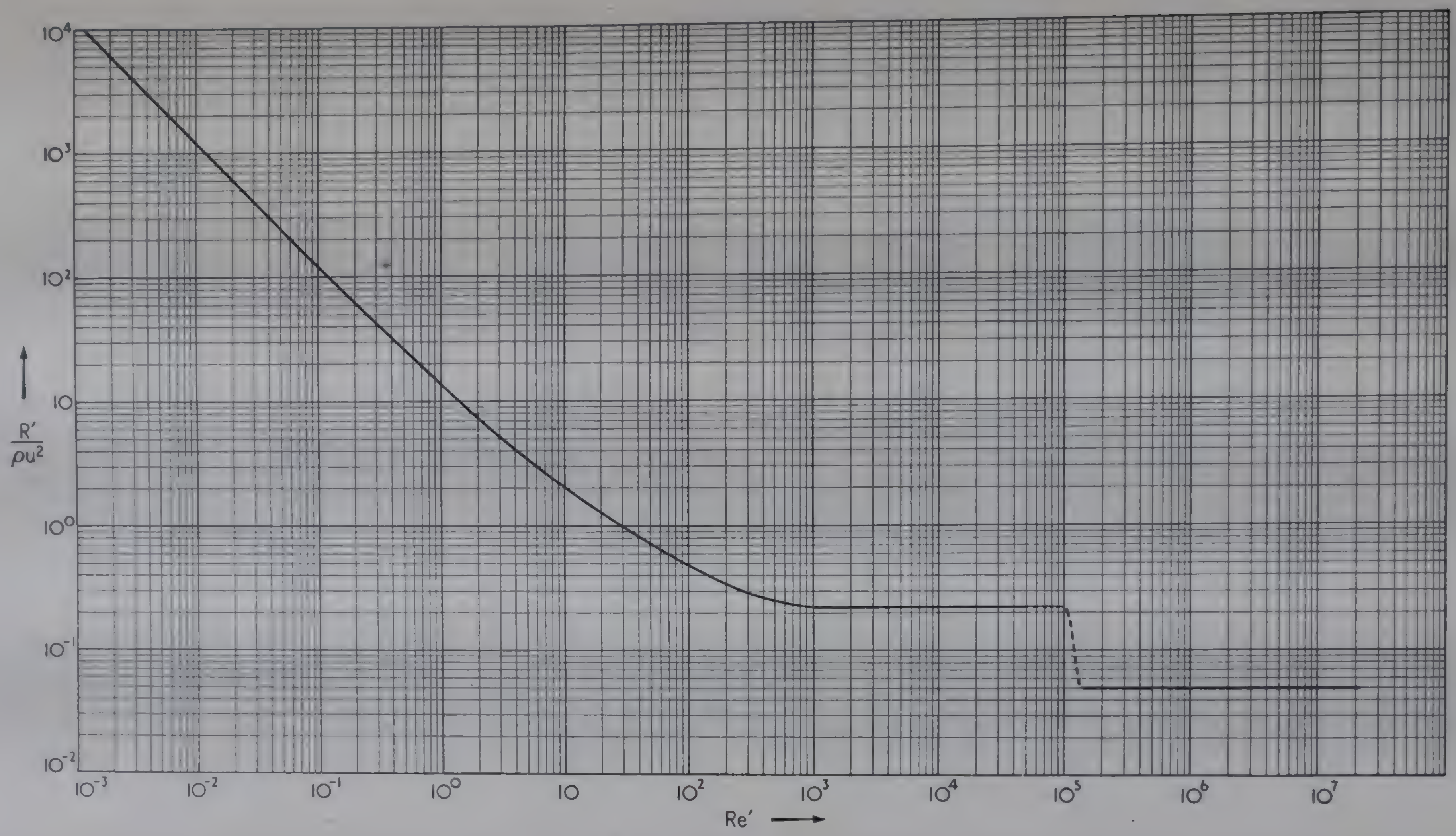


Fig. 14.5. $\frac{R'}{\rho u^2}$ versus *Re.′* for spherical particles

In region (*c*),

$$R' = 0{\cdot}22\rho u^2 \qquad \ldots.(14.11)$$

and

$$F = 0{\cdot}22\rho u^2 \,.\, \tfrac{1}{4}\pi d^2 = 0{\cdot}055\pi d^2\rho u^2 \qquad \ldots.(14.12)$$

This relation is often known as NEWTON's Law.

In region (*d*),

$$R' = 0{\cdot}05\rho u^2 \qquad \ldots.(14.13)$$

and

$$F = 0{\cdot}0125\pi d^2\rho u^2 \qquad \ldots.(14.14)$$

If a spherical particle is allowed to settle in a fluid under the action of the earth's gravitational field, it will increase in velocity until the accelerating force is exactly balanced by the resistance force. Although this state is approached exponentially, the effective acceleration period is generally of short duration for very small particles. If this terminal falling velocity is such that the corresponding value of *Re.*′ is less than 0·2, the drag force on the particle will be given by equation 14.8. If the corresponding value of *Re.*′ lies between 0·2 and 500, the drag force will be given approximately by SCHILLER and NAUMANN's equation (14.10). It should be noted, however, that if the particle has started from rest, the drag force will be given by equation 14.8 until *Re.*′ exceeds 0·2. Again if the terminal falling velocity corresponds to a value of *Re.*′ greater than about 500, the drag on the particle will be given by equation 14.12. Under terminal falling conditions, velocities are rarely high enough for *Re.*′ to approach $10^5$, with the small particles generally used in the chemical industry. The accelerating force due to gravity

$$= \tfrac{1}{6}\pi d^3(\rho_s - \rho)\,.\,g \qquad \ldots.(14.15)$$

(where $\rho_s$ is the density of the solid).

The terminal falling velocity corresponding to region (*a*) is given by

$$\tfrac{1}{6}\pi d^3(\rho_s - \rho)\,.\,g = 3\pi\mu d u$$

i.e.

$$u = \frac{d^2 g}{18\mu}(\rho_s - \rho) \qquad \ldots.(14.16)$$

The terminal falling velocity corresponding to region (*c*) is given by

$$\tfrac{1}{6}\pi d^3(\rho_s - \rho)\,.\,g = 0{\cdot}055\pi d^2\rho u^2$$

i.e.

$$u^2 = 3dg\frac{(\rho_s - \rho)}{\rho} \qquad \ldots.(14.17)$$

In the expressions which have been given for the drag force and the terminal falling velocity it has been assumed

(1) that the settling is not affected by the presence of other particles in the fluid. This condition is known as "free settling"; when the interference of other particles is appreciable, the process is known as "hindered settling".

(2) that the walls of the containing vessel do not exert an appreciable retarding effect.

(3) that the fluid can be considered as a continuous medium: i.e. that the particle is large compared with the mean free path of the molecules of the fluid: otherwise the particles may occasionally "slip" between the molecules and thus attain a velocity higher than the calculated one.

A number of small correction factors may be applied to account for the effect of the walls and for the effect of "slip." LADENBURG(8) states that the velocity of fall in a vessel of diameter $D$ must be multiplied by $\left(1 + 2{\cdot}4\,\frac{d}{D}\right)$ to give the velocity in an infinite medium. This is only approximate for values of $d/D$ approaching unity. He has introduced another similar factor, $\left(1 + 1{\cdot}7\,\frac{d}{L'}\right)$, which must be introduced when the particle is at a comparatively small distance $L'$ from the bottom of the container. According to CUNNINGHAM(9), the calculated falling velocity of particles below 0·1 micron in diameter must be multiplied by $\left(1 + \frac{J\lambda}{d}\right)$ to account for "slip," where $\lambda$ is the mean free path. DAVIES(28) gives $J$ equal to $1{\cdot}764 + 0{\cdot}562e^{-0{\cdot}785d/\lambda}$.

The behaviour of very small particles is also affected by Brownian Motion. The molecules of the fluid bombard each particle in a random manner; if the particle is small, the net resultant force acting at any instant may be large enough to cause a change in its direction of motion. This effect has been studied by DAVIES(45), who has developed an expression for the combined effects of gravitation and Brownian Motion on particles suspended in a fluid.

### Effect of Motion of the Fluid

If the fluid is moving relative to some surface other than that of the particle, there will be a superimposed velocity distribution and the drag on the particle may be altered. Thus, if the particle is situated at the axis of a vertical tube up which fluid is flowing in streamline motion, the velocity near the particle will be twice the mean velocity because of the parabolic velocity profile in the fluid. The drag force is then determined by the difference in the velocities of the fluid and the particle at the axis. On the other hand, if the motion is turbulent, the drag on the particle will normally be increased but the effect is difficult to estimate quantitatively.

If $Re.'$ is of the order of $10^5$, the drag on the sphere may be reduced if the fluid stream is turbulent. The flow in the boundary layer changes from streamline to turbulent and the formation of eddies in the wake of the particle is reduced. The higher the turbulence of the fluid, the lower is the value of $Re.'$ at which the transition from region (*c*) to region (*d*) occurs. The value of $Re.'$ at which $\frac{R'}{\rho u^2}$ is 0·15 is known as the Turbulence Number and is taken as an indication of the turbulence of the fluid.

### Terminal Falling Velocities

Expressions have already been given for the terminal falling velocities of spherical particles but each is applicable over only a limited range of values of the Reynolds Group $Re.'$. If the value of $Re.'$ is known approximately, it is possible to select the correct expression. In general, however, $Re.'$ will be unknown because it is a function of the unknown terminal velocity and a different approach must be used. The resistance force per unit projected area of the particle under terminal falling conditions is given by

$$R' \cdot \tfrac{1}{4}\pi d^2 = \tfrac{1}{6}\pi d^3 (\rho_s - \rho) \cdot g$$

i.e.

$$R' = \tfrac{2}{3} d (\rho_s - \rho) \cdot g \qquad \ldots.(14.18)$$

and

$$\frac{R'}{\rho u^2} = \frac{2dg}{3\rho u^2}(\rho_s - \rho) \qquad \ldots.(14.19)$$

TABLE 14.2. *Values of log Re.′ as a function of log $R'/\rho u^2 \cdot Re.'^2$, for spherical particles*

| log $R'/\rho u^2 \cdot Re.'^2$ | 0·0 | 0·1 | 0·2 | 0·3 | 0·4 | 0·5 | 0·6 | 0·7 | 0·8 | 0·9 |
|---|---|---|---|---|---|---|---|---|---|---|
| $\bar{2}$ | | | | | | | | $\bar{3}$·620 | $\bar{3}$·720 | $\bar{3}$·819 |
| $\bar{1}$ | $\bar{3}$·919 | $\bar{2}$·018 | $\bar{2}$·117 | $\bar{2}$·216 | $\bar{2}$·315 | $\bar{2}$·414 | $\bar{2}$·513 | $\bar{2}$·612 | $\bar{2}$·711 | $\bar{2}$·810 |
| 0 | $\bar{2}$·908 | $\bar{1}$·007 | $\bar{1}$·105 | $\bar{1}$·203 | $\bar{1}$·301 | $\bar{1}$·398 | $\bar{1}$·495 | $\bar{1}$·591 | $\bar{1}$·686 | $\bar{1}$·781 |
| 1 | $\bar{1}$·874 | $\bar{1}$·967 | 0·008 | 0·148 | 0·236 | 0·324 | 0·410 | 0·495 | 0·577 | 0·659 |
| 2 | 0·738 | 0·817 | 0·895 | 0·972 | 1·048 | 1·124 | 1·199 | 1·273 | 1·346 | 1·419 |
| 3 | 1·486 | 1·562 | 1·632 | 1·702 | 1·771 | 1·839 | 1·907 | 1·974 | 2·040 | 2·106 |
| 4 | 2·171 | 2·236 | 2·300 | 2·363 | 2·425 | 2·487 | 2·548 | 2·608 | 2·667 | 2·725 |
| 5 | 2·783 | 2·841 | 2·899 | 2·956 | 3·013 | 3·070 | 3·127 | 3·183 | 3·239 | 3·295 |

The curve of $R'/\rho u^2$ cannot be used to calculate $u$, because both $Re.'$ and $R'/\rho u^2$ are themselves functions of $u$, whereas the dimensionless group $R'/\rho u^2 \times Re.'^2$, does not involve $u$, since

$$\frac{R'}{\rho u^2} \times \frac{u^2 d^2 \rho^2}{\mu^2} = \frac{2dg\,(\rho_s - \rho)}{3\rho u^2} \times \frac{u^2 d^2 \rho^2}{\mu^2}$$

$$= \frac{2d^3 \rho g (\rho_s - \rho)}{3\mu^2} \qquad \ldots.(14.20)$$

Thus $R'/\rho u^2 \times Re.'^2$ can be evaluated if the properties of the fluid and the particle are known. In Table 14.2, values of log $Re.'$ are given as a function of log $R'/\rho u^2 \cdot Re.'^2$ and the data are represented in graphical form in Fig. 14.6 (data are taken from tables given by HEYWOOD(32)). In order to determine the terminal falling velocity of a particle, $R'/\rho u^2 \cdot Re.'^2$ is evaluated and the corresponding value of $Re.'$, and hence of the terminal velocity, is found either from Table 14.2 or from Fig. 14.6.

A similar difficulty is encountered in calculating the size of a sphere having a given terminal falling velocity, since $Re.'$ and $R'/\rho u^2$ are both functions of the diameter $d$ of the particle. This calculation is similarly facilitated by the use of another combination, $R'/\rho u^2 \times Re.'^{-1}$, which is independent of diameter.

$$\frac{R'}{\rho u^2} \times Re.'^{-1} = \frac{2\mu g}{3\rho^2 u^3}(\rho_s - \rho) \qquad \ldots.(14.21)$$

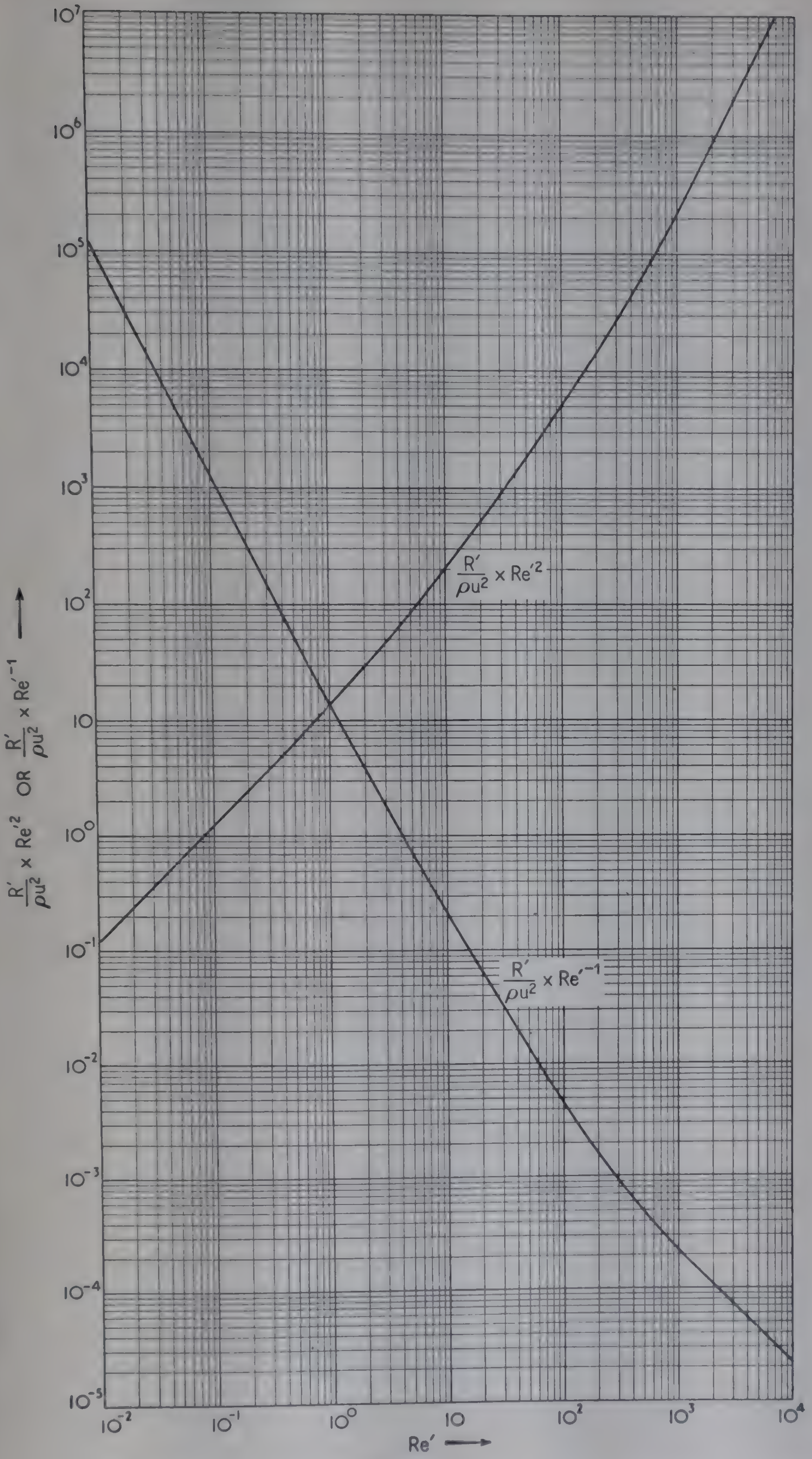


Fig. 14.6. $\frac{R'}{\rho u^2} \cdot Re.'^2$ and $\frac{R'}{\rho u^2} \cdot Re.'^{-1}$ versus *Re.′* for spherical particles

Log $Re.'$ is given as a function of $\log (R'/\rho u^2 \times Re.'^{-1})$ in Table 14.3 and the functions are plotted in Fig. 14.6. The diameter of a sphere of known terminal falling velocity can then be calculated by evaluating $R'/\rho u^2 \times Re.'^{-1}$, and then finding the corresponding value of $Re.'$, from which the diameter can be calculated.

TABLE 14.3. *Values of log Re.′ as a function of log $R'/\rho u^2 . Re.'^{-1}$ for spherical particles*

| $\log R'/\rho u^2 . Re.'^{-1}$ | 0·0 | 0·1 | 0·2 | 0·3 | 0·4 | 0·5 | 0·6 | 0·7 | 0·8 | 0·9 |
|---|---|---|---|---|---|---|---|---|---|---|
| $\bar{5}$ | | | | | | | | | | 3·401 |
| $\bar{4}$ | 3·316 | 3·231 | 3·148 | 3·065 | 2·984 | 2·903 | 2·824 | 2·745 | 2·668 | 2·591 |
| $\bar{3}$ | 2·517 | 2·443 | 2·372 | 2·300 | 2·231 | 2·162 | 2·095 | 2·027 | 1·961 | 1·894 |
| $\bar{2}$ | 1·829 | 1·763 | 1·699 | 1·634 | 1·571 | 1·508 | 1·496 | 1·383 | 1·322 | 1·260 |
| $\bar{1}$ | 1·200 | 1·140 | 1·081 | 1·022 | 0·963 | 0·904 | 0·846 | 0·788 | 0·730 | 0·672 |
| 0 | 0·616 | 0·560 | 0·505 | 0·449 | 0·394 | 0·339 | 0·286 | 0·232 | 0·178 | 0·125 |
| 1 | 0·072 | 0·019 | $\bar{1}$·969 | $\bar{1}$·919 | $\bar{1}$·865 | $\bar{1}$·811 | $\bar{1}$·760 | $\bar{1}$·708 | $\bar{1}$·656 | $\bar{1}$·605 |
| 2 | $\bar{1}$·554 | $\bar{1}$·503 | $\bar{1}$·452 | $\bar{1}$·401 | $\bar{1}$·350 | $\bar{1}$·299 | $\bar{1}$·249 | $\bar{1}$·198 | $\bar{1}$·148 | $\bar{1}$·097 |
| 3 | $\bar{1}$·047 | $\bar{2}$·996 | $\bar{2}$·946 | $\bar{2}$·895 | $\bar{2}$·845 | $\bar{2}$·794 | $\bar{2}$·744 | $\bar{2}$·694 | $\bar{2}$·644 | $\bar{2}$·594 |
| 4 | $\bar{2}$·544 | $\bar{2}$·493 | $\bar{2}$·443 | $\bar{2}$·393 | $\bar{2}$·343 | $\bar{2}$·292 | | | | |

**Example.** What will be the settling velocity of a spherical steel particle, 0·015 in. diameter, in an oil of specific gravity 0·82 and viscosity 10 centipoises? The specific gravity of steel is 7·87.

**Solution**

For a sphere
$$\left(\frac{R'}{\rho u^2}\right) . Re.'^2 = \frac{2d^3}{3\mu^2} \rho(\rho_s - \rho) . g \qquad \text{(equation 14.20)}$$

$$= \frac{2 \times 0{\cdot}015^3 \times 2{\cdot}54^3 \times 0{\cdot}82 \times 7{\cdot}05 \times 981}{3 \times 0{\cdot}1^2}$$

$$= 20{\cdot}9$$

$$\log_{10} 20{\cdot}9 = 1{\cdot}318$$

$$\therefore \quad \log_{10} Re.' = 0{\cdot}166 \quad \text{(from Table 14·2)}$$

$$\therefore \quad Re.' = 1{\cdot}466$$

$$\therefore \quad u = 1{\cdot}466 . \frac{0{\cdot}1}{0{\cdot}82 \times 0{\cdot}0381}$$

$$= \underline{\underline{4{\cdot}7 \text{ cm/sec.}}}$$

## NON-SPHERICAL PARTICLES

### Effect of Shape and Orientation on Drag

A spherical particle is unique in that it presents the same surface to the oncoming fluid whatever its orientation. For non-spherical particles on the other hand, the orientation must be specified before the drag force can be calculated. The experimental data for the drag can be correlated in the same way as for the sphere by plotting the dimensionless group $R'/\rho u^2$ against the Reynolds Group, $Re.' = \dfrac{ud'\rho}{\mu}$, using logarithmic co-ordinates, and a separate curve is obtained for each shape of particle and for each orientation. In the above groups, $R'$ is taken, as before, as the resistance force per unit area of particle, projected on to a plane perpendicular to the direction of flow. $d'$ is defined as the diameter of the circle having the same area as the projected area of the particle and is therefore a function of the orientation, as well as the shape, of the particle.

The curve for $R'/\rho u^2$ against $Re.'$ can be divided into four regions, (a), (b), (c) and (d), as before. In region (a) the flow is entirely streamline and, although no theoretical expressions have been developed for the drag on the particle, the practical data suggest that a law of the form

$$\frac{R'}{\rho u^2} = K\, Re.'^{-1} \qquad \ldots .(14.22)$$

is applicable. The constant $K$ varies somewhat according to the shape and orientation of the particle but always has a value of about 12. In this region, a particle falling freely in the fluid under the action of gravity will normally move with its longest surface nearly parallel to the direction of motion.

At higher values of $Re.'$, the linear relation between $R'/\rho u^2$ and $Re.'^{-1}$ no longer holds and the slope of the curve gradually changes until $R'/\rho u^2$ becomes independent of $Re.'$ in region (*c*). Region (*b*) represents transition conditions and commences at a lower value of $Re.'$, and a correspondingly higher value of $R'/\rho u^2$, than in the case of the sphere: a freely falling particle will tend to change its orientation as the value of $Re.'$ changes and some instability may be apparent. In region (*c*) the particle will tend to fall so that it is presenting the maximum possible surface to the oncoming fluid. Typical values of $R'/\rho u^2$ for non-spherical particles in region (*c*) are as follows.

Thin rectangular plates, arranged with their planes perpendicular to the direction of motion.

| | |
|---|---|
| Length/breadth $= 1 - 5$ | $R'/\rho u^2 = 0{\cdot}6$ |
| 20 | 0·75 |
| $\infty$ | 0·95 |

Cylinders with axes parallel to direction of motion.

| | |
|---|---|
| Length/diameter $= 1$ | $R'/\rho u^2 = 0{\cdot}45$ |

Cylinders with axes perpendicular to the direction of motion.

| | |
|---|---|
| Length/diameter $= 1$ | $R'/\rho u^2 = 0{\cdot}3$ |
| 5 | 0·35 |
| 20 | 0·45 |
| $\infty$ | 0·6 |

It should be noted that all the above values of $R'/\rho u^2$ are higher than the value of 0·22 for the sphere.

### Terminal Falling Velocities

HEYWOOD[32] has developed an approximate method for calculating the terminal falling velocity of a non-spherical particle, or for calculating its size from its terminal falling velocity. The method is an adaptation of his method for spheres.

A mean projected diameter of the particle $d_p$ is defined as the diameter of a circle having the same area as the profile of the particle when it is lying in its most stable position. HEYWOOD selected this particular dimension because it is easily measured by microscopic examination.

If $d_p$ is the mean projected diameter, the mean projected area is $\frac{1}{4}\pi d_p^2$ and the volume is $k'd_p^3$, where $k'$ is a constant whose value depends on the shape of the particle. For a spherical particle, $k'$ is equal to $\pi/6$. For rounded isometric particles (i.e. particles in which the dimension in three mutually perpendicular directions is approximately the same) $k'$ is about 0·5, but for angular particles $k'$ is about 0·4. For most minerals $k'$ lies between 0·2 and 0·5.

The method of calculating the terminal falling velocity consists in evaluating $R'/\rho u^2 \,.\, Re.'^2$, using $d_p$ as the characteristic linear dimension of the particle and $\frac{1}{4}\pi d_p^2$ as the projected area in a plane perpendicular to the direction of motion. The corresponding value of $Re.'$ is then found from Table 14.2 or Fig. 14.6, which refer to spherical particles, and a correction is then applied to the value of log $Re.'$ to account for the deviation from spherical shape. Values of this correction factor, which is a function both of $k'$ and of $R'/\rho u^2 \,.\, Re.'^2$ are given in Table 14.4. A similar procedure is adopted for calculating the size of a particle of given terminal velocity.

The method is only approximate because it is assumed that $k'$ completely defines the shape of the particle, whereas there are many different shapes of particle for which the value of $k'$ is the same. Further it assumes that the diameter $d_p$ is the same as the mean projected diameter $d'$. This is very nearly so in regions (b) and (c), but in region (a) the particle tends to settle so that the longest face is parallel to the direction of motion and some error may therefore be introduced in the calculation, as indicated by HEISS and COULL[75].

For the non-spherical particle, the total drag force

$$= R' \,.\, \tfrac{1}{4}\pi d_p^2 = (\rho_s - \rho)g \,.\, k'd_p^3$$

Thus

$$\frac{R'}{\rho u^2} = \frac{4k'd_p g}{\pi \rho u^2}(\rho_s - \rho) \quad \ldots.(14.23)$$

$$\frac{R'}{\rho u^2} \cdot Re.'^2 = \frac{4k'\rho d_p^3 g}{\mu^2 \pi}(\rho_s - \rho) \quad \ldots.(14.24)$$

and

$$\frac{R'}{\rho u^2} \cdot Re.'^{-1} = \frac{4k'\mu g}{\pi \rho^2 u^3}(\rho_s - \rho) \quad \ldots.(14.25)$$

Provided $k'$ is known, the appropriate dimensionless group can be evaluated and the terminal falling velocity, or diameter, calculated.

**TABLE 14.4.** *Logarithmic corrections to log Re′ as a function of* $R'/\rho u^2 . Re.'^2$ *for non-spherical particles*

| log $R'/\rho u^2 . Re.'^2$ | $k' = 0\cdot 4$ | $k' = 0\cdot 3$ | $k' = 0\cdot 2$ | $k' = 0\cdot 1$ |
|---|---|---|---|---|
| $\bar{2}$ | −0·022 | −0·002 | +0·032 | +0·131 |
| $\bar{1}$ | −0·023 | −0·003 | +0·030 | +0·131 |
| 0 | −0·025 | −0·005 | +0·026 | +0·129 |
| 1 | −0·027 | −0·010 | +0·021 | +0·122 |
| 2 | −0·031 | −0·016 | +0·012 | +0·111 |
| 2·5 | −0·033 | −0·020 | 0·000 | +0·080 |
| 3 | −0·038 | −0·032 | −0·022 | +0·025 |
| 3·5 | −0·051 | −0·052 | −0·056 | −0·040 |
| 4 | −0·068 | −0·074 | −0·089 | −0·098 |
| 4·5 | −0·083 | −0·093 | −0·114 | −0·146 |
| 5 | −0·097 | −0·110 | −0·135 | −0·186 |
| 5·5 | −0·109 | −0·125 | −0·154 | −0·224 |
| 6 | −0·120 | −0·134 | −0·172 | −0·255 |

**TABLE 14.5.** *Logarithmic corrections to log Re.′ as a function of* $R'/\rho u^2 . Re.'^{-1}$ *for non-spherical particles*

| log $R'/\rho u^2 . Re.'^{-1}$ | $k' = 0\cdot 4$ | $k' = 0\cdot 3$ | $k' = 0\cdot 2$ | $k' = 0\cdot 1$ |
|---|---|---|---|---|
| $\bar{4}$ | +0·185 | +0·217 | +0·289 | |
| $\bar{4}\cdot 5$ | +0·149 | +0·175 | +0·231 | |
| $\bar{3}$ | +0·114 | +0·133 | +0·173 | +0·282 |
| $\bar{3}\cdot 5$ | +0·082 | +0·095 | +0·119 | +0·170 |
| $\bar{2}$ | +0·056 | +0·061 | +0·072 | +0·062 |
| $\bar{2}\cdot 5$ | +0·038 | +0·034 | +0·033 | −0·018 |
| $\bar{1}$ | +0·028 | +0·018 | +0·007 | −0·053 |
| $\bar{1}\cdot 5$ | +0·024 | +0·013 | −0·003 | −0·061 |
| 0 | +0·022 | +0·011 | −0·007 | −0·062 |
| 1 | +0·019 | +0·009 | −0·008 | −0·063 |
| 2 | +0·017 | +0·007 | −0·010 | −0·064 |
| 3 | +0·015 | +0·005 | −0·012 | −0·065 |
| 4 | +0·013 | +0·003 | −0·013 | −0·066 |
| 5 | +0·012 | +0·002 | −0·014 | −0·066 |

**Example.** What will be the settling velocities of mica plates, $\frac{1}{32}$ in. thick and ranging in area from 0·01 to 1 sq in., in an oil of specific gravity 0·82 and viscosity 10 centipoises? The specific gravity of mica is 3·0.

**Solution**

| | smallest particles | largest particles |
|---|---|---|
| $A'$ | 0·01 sq in. | 1 sq in. |
| $d_p$ | $\sqrt{\frac{0{\cdot}04}{\pi}} = 0{\cdot}113$ in. | $\sqrt{\frac{4}{\pi}} = 1{\cdot}13$ in. |
| $d_p^3$ | 0·00144 cu in. | 1·44 cu in. |
| volume | 0·00031 cu in. | 0·031 cu in. |
| $k'$ | 0·22 | 0·022 |

$$\left(\frac{R'}{\rho u^2}\right) . Re.'^2 = \frac{4k'}{\mu^2\pi}(\rho_s - \rho)\rho d_p^3 g \qquad \text{....(14.24)}$$

$$= \frac{4 \times 0{\cdot}22}{0{\cdot}1^2 . \pi}(3{\cdot}0 - 0{\cdot}82) \times 0{\cdot}82 \times 0{\cdot}00144 \times 2{\cdot}54^3 \times 981$$

$$= 1160 \text{ for smallest particle}$$

and 116,000 for largest particle.

| | smallest particle | largest particle | |
|---|---|---|---|
| $\log \frac{R'}{\rho u^2} . Re.'^2$ | 3·064 | 5·064 | |
| $\log Re.'$ | 1·537 | 2·820 | (from Table 14.2) |
| Correction from Table 14·4 | −0·029 | −0·300 | (estimated) |
| Corrected $\log Re.'$ | 1·508 | 2·520 | |
| $Re.'$ | 32·2 | 330 | |
| $u$ | 13·7 cm/sec | 13·5 cm/sec | |

Thus it is seen that all the mica particles settle at approximately the same velocity.

## MOTION OF BUBBLES AND DROPS

The drag force acting on a gas bubble or a liquid droplet will not, in general, be the same as that acting on a rigid particle of the same shape and size because circulating currents are set up inside the bubble. The velocity gradient at the surface is thereby reduced and the drag force is therefore less than for the rigid particle. If the drop is very small, however, surface tension forces prevent the circulation of the fluid and the drag is unaffected. HADAMARD(10) showed that, if the effects of surface energy are neglected, the terminal falling velocity of a drop, as calculated from STOKES' Law, must be multiplied by a factor $Q$, to account for the internal circulation, such that

$$Q = \frac{3\mu + 3\mu_l}{2\mu + 3\mu_l} \qquad \text{....(14.26)}$$

where $\mu$ is the viscosity of the continuous fluid and $\mu_l$ is the viscosity of the fluid forming the drop or bubble. This expression applies only in the range for which STOKES' Law is valid.

If $\frac{\mu_l}{\mu}$ is large, $Q$ approaches unity, but if $\frac{\mu_l}{\mu}$ is small, $Q$ approaches a value of 1·5. Thus the effect of circulation is small when a liquid drop falls in a gas but is large when a gas bubble rises in a liquid. If the fluid within the drop is very viscous, the amount of energy which has to be transferred in order to induce circulation is large and circulation effects are therefore small.

Drops and bubbles are, in addition, subject to deformation because of the differences in the pressures acting on various parts of the surface. Thus, when a drop is settling in a still fluid, both the hydrostatic and the impact pressures will be greater on the forward face than on the rear face and will tend to flatten the drop, whereas the viscous drag will tend to elongate it. Deformation of the drop is opposed by the surface tension forces so that very small drops retain their spherical shape, whereas large drops may be considerably deformed and the resistance to their motion thereby increased. For drops above a certain size, the deformation is so great that the drag force increases at the same rate as the volume, and the terminal falling velocity therefore becomes independent of size.

## TWO DIMENSIONAL MOTION OF A PARTICLE IN A FLUID

Consider the motion of a particle of mass $m$ in the earth's gravitational field. At some time $t$ the particle will be moving at an angle $\alpha$ to the horizontal with a velocity $u$ (Fig. 14.7). This velocity $u$ can then be resolved into two components, $\dot{x}$ and $\dot{y}$, in the horizontal and vertical directions. ($\dot{x}$ and $\ddot{x}$ will be taken to denote the first and second derivatives of the displacement $x$ in the $X$-direction with respect to time, and $\dot{y}$ and $\ddot{y}$ the corresponding derivatives of $y$.)

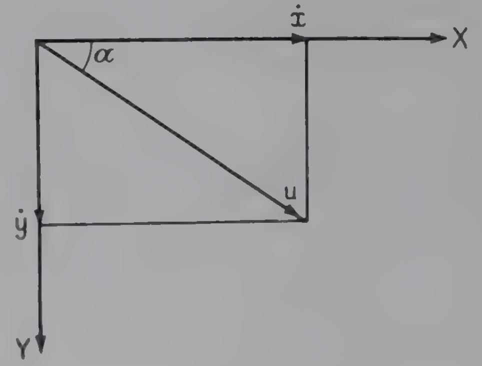


Fig. 14.7.
Two dimensional motion of particle

Now $$\cos\alpha = \frac{\dot{x}}{u} \qquad \ldots.(14.27)$$

$$\sin\alpha = \frac{\dot{y}}{u} \qquad \ldots.(14.28)$$

and $$u = \sqrt{\dot{x}^2 + \dot{y}^2} \qquad \ldots.(14.29)$$

There are two forces acting on the body

(1) In the vertical direction, the apparent weight of the particle, $mg\left(1 - \frac{\rho}{\rho_s}\right)$.

(2) The drag force which is equal to $R'A'$ and acts in such a direction as to oppose the motion of the particle. Its direction therefore changes as $\alpha$ changes. Here $A'$ is the projected area of the particle on a plane at right angles to the direction of motion and its value varies with the orientation of the particle in the fluid. The drag force can be expressed by the relation

$$F = \frac{R'}{\rho u^2} \times \rho u^2 \times A' \qquad \ldots.(14.30)$$

This has a component in the $X$-direction of

$$\frac{R'}{\rho u^2} \times \rho u^2 A' \cos \alpha = \frac{R'}{\rho u^2} A' \rho \dot{x} \sqrt{\dot{x}^2 + \dot{y}^2}$$

and in the $Y$-direction

$$\frac{R'}{\rho u^2} \times \rho u^2 A' \sin \alpha = \frac{R'}{\rho u^2} \times A' \rho \dot{y} \sqrt{\dot{x}^2 + \dot{y}^2}$$

The equations of motion in the $X$- and $Y$-directions are therefore

$$m\ddot{x} = -\frac{R'}{\rho u^2} \rho A' . \dot{x} \sqrt{\dot{x}^2 + \dot{y}^2} \qquad \text{....(14.31)}$$

and

$$m\ddot{y} = -\frac{R'}{\rho u^2} \rho A' . \dot{y} \sqrt{\dot{x}^2 + \dot{y}^2} + mg\left(1 - \frac{\rho}{\rho_s}\right) \qquad \text{....(14.32)}$$

assuming the velocities $\dot{x}$ and $\dot{y}$ to be positive. If $\dot{x}$ or $\dot{y}$ is negative the drag force acts in the opposite direction and the corresponding term bears a positive sign. In general direct integration is not possible because the equation of motion in the $X$-direction involves the velocity in the $Y$-direction and vice versa, but for the following cases we can obtain a solution,

(1) Flow under conditions where form drag is negligible,
(2) Unidimensional flow, when the effect of gravity can be neglected, and
(3) Unidimensional flow in the vertical direction, under the action of gravity.

**Motion of a Particle (Form Drag Negligible).**

Under these conditions, $R'/\rho u^2$ is given by the relation

$$\frac{R'}{\rho u^2} = K . Re.'^{-1} \qquad \text{....(14.22)}$$

If this value is substituted in equations (14.31) and (14.32), we have

$$\ddot{x} = -\frac{A'\dot{x}\rho}{m} \cdot \frac{K\mu}{ud'\rho} \sqrt{\dot{x}^2 + \dot{y}^2} = -\frac{K\mu}{md'} A'\dot{x} \qquad \text{....(14.33)}$$

and

$$\ddot{y} = -\frac{K\mu}{md'} A' . \dot{y} + g . \left(\frac{\rho_s - \rho}{\rho_s}\right) \qquad \text{....(14.34)}$$

since $u = \sqrt{\dot{x}^2 + \dot{y}^2}$. (equation 14.29)

Thus, in this particular case, the motions in two mutually perpendicular directions can be considered independently. Further, it is seen that if $\dot{x}$ or $\dot{y}$ is negative, the resistance term becomes positive and the equation of motion is still valid.

For the motion of a spherical particle, $K = 12$, so that

$$\ddot{x} = -\frac{\frac{\pi}{4} d^2 \,.\, 12\mu}{\frac{\pi}{6} d^3 \rho_s d} \dot{x}$$

$$= -18 \frac{\mu}{d^2 \rho_s} \dot{x} \qquad \ldots.(14.35)$$

$$= -a\dot{x} \qquad \ldots.(14.36)$$

and

$$\ddot{y} = -\frac{\frac{\pi}{4} d^2 \,.\, 12\mu}{\frac{\pi}{6} d^3 \rho_s d} \dot{y} + \left(1 - \frac{\rho}{\rho_s}\right) . g$$

$$= -18 \frac{\mu}{d^2 \rho_s} \dot{y} + \left(1 - \frac{\rho}{\rho_s}\right) . g \qquad \ldots.(14.37)$$

$$= -a\dot{y} + b \qquad \ldots.(14.38)$$

(where $$a = 18 \frac{\mu}{d^2 \rho_s} \qquad \ldots.(14.39)$$

and $$b = \left(1 - \frac{\rho}{\rho_s}\right) . g \qquad \ldots.(14.40)$$

are constant for a given fluid and particle).

These equations can now be integrated independently.

$$\ddot{y} = -a\dot{y} + b \qquad \ldots.(14.38)$$

Integrating with respect to $t$,

$$\dot{y} = -ay + bt + \text{constant.}$$

The axes will now be so chosen that the particle is at the origin at time, $t = 0$. Let the initial component of the velocity of the particle in the $Y$-direction be $v$. Thus, when $t = 0$, $y = 0$ and $\dot{y} = v$, and the constant $= v$.

i.e. $$\dot{y} + ay = bt + v$$

$\therefore$ $$e^{at} . \dot{y} + e^{at} . ay = (bt + v)\, e^{at}$$

Thus $$e^{at} . y = (bt + v) . \frac{e^{at}}{a} - \int b . \frac{e^{at}}{a} \mathrm{d}t$$

$$= (bt + v) . \frac{e^{at}}{a} - \frac{b}{a^2} e^{at} + \text{constant}$$

When $t = 0$, $y = 0$, and the constant $= \frac{b}{a^2} - \frac{v}{a}$

Thus $$y = \frac{b}{a} t + \frac{v}{a} - \frac{b}{a^2} + \left(\frac{b}{a^2} - \frac{v}{a}\right) e^{-at} \qquad \ldots.(14.41)$$

$$\left(\text{where } a = 18 \frac{\mu}{d^2 \rho_s} \quad \text{and} \quad b = \left(1 - \frac{\rho}{\rho_s}\right) g\right)$$

This equation enables the displacement of the particle in the $Y$-direction to be calculated at any time $t$. For the displacement in the $X$-direction

$$\ddot{x} = -a\dot{x} \qquad \ldots.(14.36)$$

$$\therefore \qquad \dot{x} = -ax + \text{constant}$$

Let $w$ be the component of the velocity of the particle in the $X$-direction at time, $t = 0$.

Then, when $t = 0$, $x = 0$ and $\dot{x} = w$.

The constant therefore is equal to $w$.

Hence $$\dot{x} + ax = w$$

$$\therefore \qquad x \,.\, e^{at} = \frac{w}{a} \,.\, e^{at} + \text{constant.}$$

When $t = 0$, $x = 0$, and the constant $= -w/a$,

$$\therefore \qquad x \,.\, e^{at} = \frac{w}{a}(e^{at} - 1)$$

i.e. $$x = \frac{w}{a}(1 - e^{-at}) \qquad \ldots.(14.42)$$

$$\left(\text{where } a = \frac{18\mu}{d^2\rho_s}\right)$$

Thus the displacement in the $X$-direction can also be calculated for any time $t$.

By eliminating $t$ between equations (14.41) and (14.42), a relation between the displacements in the $X$- and $Y$-directions is obtained. Equations of this form are useful for calculating the trajectories of particles in size-separation equipment.

From equation 14.42, $$e^{-at} = 1 - \frac{ax}{w}$$

and $$t = -\frac{1}{a}\ln\left(1 - \frac{ax}{w}\right) \qquad \ldots.(14.43)$$

Substitution in equation (14.41) gives

$$y = \frac{b}{a} \times -\frac{1}{a}\ln\left(1 - \frac{ax}{w}\right) + \frac{v}{a} - \frac{b}{a^2} + \left(\frac{b}{a^2} - \frac{v}{a}\right)\left(1 - \frac{ax}{w}\right)$$

$$= -\frac{b}{a^2}\ln\left(1 - \frac{ax}{w}\right) - \frac{bx}{aw} + \frac{vx}{w} \qquad \ldots.(14.44)$$

The values of $a$ and $b$ can now be substituted and the final relation is

$$y = -\frac{g\rho_s(\rho_s - \rho)d^4}{324\mu^2}\left\{\ln\left(1 - \frac{18\mu x}{w\rho_s d^2}\right) + \frac{18\mu x}{w\rho_s d^2}\left(1 - \frac{18v\mu}{d^2(\rho_s - \rho)g}\right)\right\}$$

$$\ldots.(14.45)$$

### Unidimensional Motion in the Absence of Gravitation

When the resistance force is very large compared with the gravitational force, the latter may be neglected. The only force then acting on the body is that due to the resistance of the fluid and the direction of motion will not alter. This condition is met when the densities of the particle and of the fluid are almost the same, when the particle is very small and when the viscosity of the fluid and the relative velocity are high.

Consider the motion of a particle in the $X$-direction under conditions where the effect of gravity can be neglected. Since $\dot{y} = 0$, we have from equation 14.31, for $\dot{x}$ positive,

$$m\ddot{x} = -\frac{R'}{\rho u^2} A' . \rho \dot{x}^2 \qquad \ldots.(14.46)$$

For a sphere,

$$\ddot{x} = -\frac{R'}{\rho u^2} \cdot \frac{\frac{1}{4}\pi d^2 \rho}{\frac{1}{6}\pi d^3 \rho_s} \dot{x}^2$$

$$= -\frac{R'}{\rho u^2} \cdot \frac{1{\cdot}5\rho}{d\rho_s} \dot{x}^2 \qquad \ldots.(14.47)$$

***Resistance due to Skin Friction only***. Under these conditions of motion,

$$\frac{R'}{\rho u^2} = 12\, Re.'^{-1} \qquad \ldots.(14.2)$$

and

$$\ddot{x} = -\frac{18\mu}{d^2 \rho_s} \dot{x} \qquad \ldots.(14.35)$$

This equation has already been integrated to give

$$x = \frac{w}{a}(1 - e^{-at}) \qquad \ldots.(14.42)$$

$$\left(\text{where } a = \frac{18\mu}{d^2 \rho_s} \text{ and } w \text{ is the initial velocity}\right)$$

Equation 14.42 is applicable if $Re.'$ is less than about 0·2, and whether $w$ is positive or negative.

***Resistance due to Form Drag only***. If $Re.'$ exceeds about 500, $R'/\rho u^2$ attains a constant value of 0·22.

Then

$$\ddot{x} = -\frac{0{\cdot}33}{d} \frac{\rho}{\rho_s} \dot{x}^2 \qquad \ldots.(14.48)$$

$$= -c\dot{x}^2 \qquad \ldots.(14.49)$$

where

$$c = \frac{0{\cdot}33}{d} \frac{\rho}{\rho_s} \qquad \ldots.(14.50)$$

Thus

$$\frac{\mathrm{d}\dot{x}}{\dot{x}^2} = -c\,.\,\mathrm{d}t.$$

Integrating,

$$-\dot{x}^{-1} = -ct + \text{constant.}$$

When $t = 0$, $\dot{x} = w$, and therefore the constant $= -w^{-1}$

Thus
$$\dot{x}^{-1} = ct + w^{-1}$$

$\therefore$
$$\frac{\mathrm{d}t}{ct + w^{-1}} = \mathrm{d}x$$

and
$$x = \frac{1}{c} \ln \left( t + \frac{1}{wc} \right) + \text{constant}$$

When $t = 0$, $x = 0$ and the constant $= -\dfrac{1}{c} \ln \dfrac{1}{wc}$

Thus
$$x = \frac{1}{c} \ln \frac{t + \dfrac{1}{wc}}{\dfrac{1}{wc}}$$

$$= \frac{1}{c} \ln (wct + 1) \qquad \ldots .(14.51)$$

$$\left( \text{where } c = \frac{0{\cdot}33\rho}{d\rho_s} \right)$$

If $w$ is negative,
$$x = -\frac{1}{c} \ln (1 - wct) \qquad \ldots .(14.52)$$

***Transition Region.*** When $Re.'$ lies between about 0.2 and 500, $R'/\rho u^2$ cannot readily be expressed as a simple function of $Re'$. A graphical solution of the problem is therefore necessary.

For a spherical particle, we have, for $w$ positive,
$$\ddot{x} = -\frac{R'}{\rho u^2} \times \frac{1{\cdot}5}{d} \frac{\rho}{\rho_s} \dot{x}^2 \qquad \ldots .(14.47)$$

Now
$$\dot{x} = Re.' \,.\, \frac{\mu}{d\rho}$$

and thus
$$\frac{\mu}{d\rho} \frac{\mathrm{d}Re.'}{\mathrm{d}t} = -\left( \frac{R'}{\rho u^2} \right) \times \frac{1{\cdot}5}{d} \frac{\rho}{\rho_s} \frac{\mu^2}{d^2 \rho^2} Re.'^2$$

i.e.
$$\frac{\mathrm{d}Re.'}{\mathrm{d}t} = -\frac{1{\cdot}5\mu}{d^2 \rho_s} \times \left( \frac{R'}{\rho u^2} \right) . Re.'^2$$

so that
$$t = -\frac{2d^2 \rho_s}{3\mu} \int_{Re.'_1}^{Re.'_2} \frac{1}{(R'/\rho u^2) \,.\, Re.'^2} \,\mathrm{d}\, Re.' \qquad \ldots .(14.53)$$

If $w$ is negative,
$$t = +\frac{2d^2 \rho_s}{3\mu} \int_{Re.'_1}^{Re.'_2} \frac{1}{\left( \dfrac{R'}{\rho u^2} \right) . \, Re.'^2} \,.\, \mathrm{d}\, Re' \qquad \ldots .(14.54)$$

This expression can be integrated graphically between the limits $Re.'_1$ and $Re.'_2$ in order to obtain $t$ as a function of $Re.'$. The corresponding values of $\dot{x}$ are then calculated; $x$ is then obtained in terms of $t$ by means of a further graphical integration. This method can be used over any range of values of $Re.'$ but, since graphical integrations are rather tedious, it is better to use the analytical expressions already derived for conditions corresponding to $Re.'$ below 0·2, or greater than 500, and to confine the application of the graphical method to the intervening region. SCHILLER and NAUMANN's equation (14.4) can be used for calculating the values of $\frac{R'}{\rho u^2}$ required for the graphical integration.

### Vertical Motion of a Particle

For vertical motion, the equations will be different for upward and downward motion. If the particle is initally moving downwards (taken as $y$ positive), the gravitational force and the fluid resistance will act in opposite directions; the particle will then always move in the same sense. On the other hand if the particle is initally moving upwards, the gravitational and resistance forces will both act downwards so that, in due course, it will be brought to rest and will then move in the opposite sense. As the equations of motion are different for the two senses, care must be exercised in the application of the expressions which are obtained.

Consider the equation of motion for a particle moving vertically downwards. From equation (14.32),

$$m\ddot{y} = -\frac{R'}{\rho u^2} \cdot A' \cdot \rho \dot{y}^2 + mg\left(1 - \frac{\rho}{\rho_s}\right) \qquad \ldots\ldots(14.55)$$

i.e.

$$\ddot{y} = -\frac{R'}{\rho u^2} \cdot \frac{A'}{m} \cdot \rho \dot{y}^2 + g\left(1 - \frac{\rho}{\rho_s}\right) \qquad \ldots\ldots(14.56)$$

For a spherical particle, $A' = \frac{1}{4}\pi d^2$ and $m = \frac{1}{6}\pi d^3 \rho_s$.

Thus

$$\ddot{y} = -\frac{R'}{\rho u^2} \times \frac{1\cdot5}{d} \cdot \frac{\rho}{\rho_s} \dot{y}^2 + g\left(1 - \frac{\rho}{\rho_s}\right) \qquad \ldots\ldots(14.57)$$

***Resistance due to Skin Friction only.*** Under these conditions of motion,

$$\frac{R'}{\rho u^2} = 12\, Re.'^{-1} \qquad \ldots\ldots(14.2)$$

and

$$\ddot{y} = -\frac{18}{d^2 \rho_s} \mu \cdot \dot{y} + g\left(1 - \frac{\rho}{\rho_s}\right) \qquad \ldots\ldots(14.58)$$

This equation, which is applicable whether the particle is moving upwards or downwards, has already been integrated to give

$$y = \frac{b}{a} t + \frac{v}{a} - \frac{b}{a^2} + \left(\frac{b}{a^2} - \frac{v}{a}\right) \cdot e^{-at} \qquad \ldots\ldots(14.41)$$

$\left(\text{where } a = 18\,\frac{\mu}{d^2 \rho_s} \text{ and } b = \left(1 - \frac{\rho}{\rho_s}\right) \cdot g\right)$

***Resistance due to Form Drag only.*** When the value of *Re.'* exceeds about 500,

$$\frac{R'}{\rho u^2} \doteq 0{\cdot}22. \qquad \ldots.(14.51)$$

Thus

$$\ddot{y} = -\frac{0{\cdot}33}{d}\frac{\rho}{\rho_s}\dot{y}^2 + g\left(1 - \frac{\rho}{\rho_s}\right) \qquad \ldots.(14.59)$$

$$= -c\dot{y}^2 + b \qquad \ldots.(14.60)$$

where

$$c = \frac{0{\cdot}33}{d}\frac{\rho}{\rho_s} \qquad \ldots.(14.50)$$

and

$$b = g\left(1 - \frac{\rho}{\rho_s}\right) \qquad \ldots.(14.40)$$

If the particle is moving upwards, $\dot{y}$ is negative and the drag force is positive. Thus, for this condition,

$$\ddot{y} = c\dot{y}^2 + b \qquad \ldots.(14.61)$$

Equations (14.60) and (14.61) will now be integrated separately.

*Motion downwards:—*

$$\ddot{y} = -c\dot{y}^2 + b \qquad \ldots.(14.60)$$

$$\therefore \qquad \frac{\mathrm{d}\dot{y}}{b - c\dot{y}^2} = \mathrm{d}t$$

$$\therefore \qquad \frac{\mathrm{d}\dot{y}}{f^2 - \dot{y}^2} = c\,.\,\mathrm{d}t$$

where

$$f = \sqrt{\frac{b}{c}} = \sqrt{\frac{d(\rho_s - \rho)\,.\,g}{0{\cdot}33\rho}} \qquad \ldots.(14.62)$$

Integrating,

$$\frac{1}{2f}\ln\frac{f + \dot{y}}{f - \dot{y}} = ct + \text{constant}$$

When $t = 0$, $\dot{y} = v$, say, and therefore the constant $= \dfrac{1}{2f}\ln\dfrac{f + v}{f - v}$

$$\therefore \qquad \frac{1}{2f}\ln\left(\frac{f + \dot{y}}{f - \dot{y}}\right)\left(\frac{f - v}{f + v}\right) = ct$$

$$\therefore \qquad \left(\frac{f + \dot{y}}{f - \dot{y}}\right)\left(\frac{f - v}{f + v}\right) = e^{2fct}$$

$$\therefore \qquad -1 + \frac{2f}{f - \dot{y}} = \frac{f + v}{f - v}e^{2fct}$$

$$\therefore \qquad f - \dot{y} = \frac{2f}{1 + \dfrac{f + v}{f - v}\,.\,e^{2fct}}$$

$$\therefore \qquad y = ft - 2f\int\frac{\mathrm{d}t}{1 + \dfrac{f + v}{f - v}\,.\,e^{2fct}}$$

$$= ft - 2f\,.\,I \quad \text{(say)}$$

$$I = \int\frac{\mathrm{d}t}{1 + j\,.\,e^{pt}} \left(\text{where } j = \frac{f + v}{f - v} \text{ and } p = 2fc\right)$$

Put $s = 1 + j\,.\,e^{pt}$

Then
$$ds = pj \,.\, e^{pt}\,dt = p\,.\,(s-1)\,.\,dt$$

Thus
$$\begin{aligned} I &= \int \frac{ds}{p\,.\,s\,.\,(s-1)} \\ &= \frac{1}{p}\int\left\{\frac{1}{s-1} - \frac{1}{s}\right\} ds \\ &= \frac{1}{p}\ln\frac{s-1}{s} + \text{constant} \\ &= \frac{1}{p}\ln\frac{j\,.\,e^{pt}}{j\,.\,e^{pt}+1} + \text{constant} \\ &= \frac{1}{2fc}\ln\frac{1}{1+\dfrac{f-v}{f+v}\,.\,e^{-2fct}} + \text{constant} \end{aligned}$$

Thus
$$y = ft - \frac{1}{c}\ln\frac{1}{1+\dfrac{f-v}{f+v}\,.\,e^{-2fct}} + \text{constant}$$

When $t = 0$, $y = 0$ and therefore

$$\text{constant} = \frac{1}{c}\ln\frac{1}{1+\dfrac{f-v}{f+v}} = \frac{1}{c}\ln\frac{f+v}{2f}$$

∴
$$y = ft + \frac{1}{c}\ln\frac{f+v}{2f}\left\{1 + \frac{f-v}{f+v}e^{-2fct}\right\}$$

i.e.
$$y = ft + \frac{1}{c}\ln\frac{1}{2f}\{f+v+(f-v)\,.\,e^{-2fct}\} \qquad \text{....(14.63)}$$

where
$$c = \frac{0{\cdot}33}{d}\,.\,\frac{\rho}{\rho_s} \qquad \text{....(14.50)}$$

and
$$f = \sqrt{\frac{d(\rho_s-\rho)\cdot g}{0{\cdot}33\rho}} \qquad \text{....(14.62)}$$

*Upward Motion:—*

$$\ddot y = c\dot y^2 + b \qquad \text{....(14.61)}$$

∴
$$\frac{d\dot y}{b + c\dot y^2} = dt$$

∴
$$\frac{d\dot y}{f^2 + \dot y^2} = c\,.\,dt$$

where
$$f = \sqrt{\frac{b}{c}} = \sqrt{\frac{d\,.\,(\rho_s-\rho)\cdot g}{0{\cdot}33\rho}} \qquad \text{....(14.62)}$$

Integrating,

$$\frac{1}{f}\tan^{-1}\frac{\dot y}{f} = ct + \text{constant}$$

When $t = 0$, $\dot y = v$, say, and the constant $= \frac{1}{f}\tan^{-1}\frac{v}{f}$; in this case, $v$ is a negative quantity.

Thus
$$\frac{1}{f}\tan^{-1}\frac{\dot{y}}{f} = ct + \frac{1}{f}\tan^{-1}\frac{v}{f}$$

$$\therefore \qquad \frac{\dot{y}}{f} = \tan\left(fct + \tan^{-1}\frac{v}{f}\right)$$

$$\therefore \qquad y = \frac{f}{-fc}\ln\cos\left(fct + \tan^{-1}\frac{v}{f}\right) + \text{constant}$$

When $t = 0$, $y = 0$, so that
$$\text{constant} = \frac{1}{c}\ln\cos\tan^{-1}\frac{v}{f}$$

$$\therefore \qquad y = -\frac{1}{c}\ln\frac{\cos\left(fct + \tan^{-1}\frac{v}{f}\right)}{\cos\tan^{-1}\frac{v}{f}}$$

$$= -\frac{1}{c}\ln\frac{\cos fct \,.\, \cos\tan^{-1}\frac{v}{f} - \sin fct \,.\, \sin\tan^{-1}\frac{v}{f}}{\cos\tan^{-1}\frac{v}{f}}$$

i.e.
$$y = -\frac{1}{c}\ln\left(\cos fct - \frac{v}{f}\sin fct\right) \qquad \ldots.(14.64)$$

where
$$c = \frac{0{\cdot}33}{d}\frac{\rho}{\rho_s} \qquad \ldots.(14.50)$$

and
$$f = \sqrt{\frac{d(\rho_s - \rho)g}{0{\cdot}33\rho}} \qquad \ldots.(14.62)$$

***Transition Region.*** The relation between $y$ and $t$ can also be obtained graphically, though the process is more tedious than that of using the analytical solution, appropriate to the particular case in question. When $Re.'$ lies between 0·2 and 500, there is no analytical solution to the problem and the graphical method must be used.

When the spherical particle is moving downwards, i.e. when its velocity is positive,

$$\ddot{y} = -\frac{3}{2d}\times\frac{\rho}{\rho_s}\left(\frac{R'}{\rho u^2}\right)\dot{y}^2 + \left(1 - \frac{\rho}{\rho_s}\right).g \qquad \ldots.(14.57)$$

$$\therefore \qquad \frac{\mu}{\rho d}\frac{\mathrm{d}Re.'}{dt} = -\frac{3}{2d}\frac{\rho}{\rho_s}\frac{R'}{\rho u^2}Re.'^2 . \frac{\mu^2}{d^2\rho^2} + \left(1 - \frac{\rho}{\rho_s}\right).g$$

$$\therefore \qquad t = \int_{Re.'_1}^{Re.'_2}\frac{\mathrm{d}Re.'}{\dfrac{d\rho(\rho_s - \rho)g}{\mu\rho_s} - \dfrac{3\mu}{2d^2\rho_s}\cdot\dfrac{R'}{\rho u^2}\cdot Re.'^2} \qquad \ldots.(14.65)$$

If the particle is moving upwards, the corresponding expression for $t$ is

$$t = \int_{Re.'_1}^{Re.'_2}\frac{\mathrm{d}Re.'}{\dfrac{d\rho(\rho_s - \rho)g}{\mu\rho_s} + \dfrac{3\mu}{2d^2\rho_s}\dfrac{R'}{\rho u^2}\cdot Re.'^2} \qquad \ldots.(14.66)$$

From the above equations, *Re.′* can be obtained as a function of $t$. The velocity $\dot{y}$ can then be calculated. By means of a second graphical integration, the displacement $y$ can be found at any time $t$.

In using the various relations which have been obtained, it must be remembered that the law of motion of the particle will change as the relative velocity between the particle and the fluid changes.

If, for instance, a particle is initially moving upwards with a velocity $v$, so that the corresponding value of *Re.′* is greater than about 500, the relation between $y$ and $t$ will be given by equation 14.64. The velocity of the particle will progressively decrease and, when *Re′* is less than 500, the motion is obtained by application of equation 14.66. The upward velocity will then fall still further until *Re.′* falls below 0·2. While the particle is moving under these conditions, its velocity will fall to zero and will then gradually increase in the downward direction. The same equation (14.41) can be applied for the whole of the time the Reynolds group is less than 0·2, irrespective of sense. Then for higher downward velocities, the particle motion is given by equations 14.65 and 14.63.

Unidimensional motion in the vertical direction, under the action of gravity, occurs frequently in elutriation and other size separation equipment. This equipment will be described in Chapter 25.

**Example.** A material of specific gravity 2·5 is fed to a size separation plant where the separating fluid is water which rises with a velocity of 4 f.p.s. The upward vertical component of the velocity of the particles is 20 f.p.s. How far will an approximately spherical particle, ¼ in. diameter, rise relative to the walls of the plant before it comes to rest in the fluid?

**Solution.** Initial velocity of particle relative to fluid, $v = 16$ f.p.s.

$$Re.' = \frac{16 \times 30{\cdot}48 \times \frac{1}{4} \times 2{\cdot}54 \times 1}{0{\cdot}01}$$

$$= 16 \times 1930 = 30{,}800$$

When the particle has been retarded to such a velocity that $Re.' = 500$, the minimum value for which equation 14.64 is applicable,

$$\dot{y} = 0{\cdot}259 \text{ f.p.s.} = 7{\cdot}9 \text{ cm/sec.}$$

When *Re.′* is greater than 500, the relation between the displacement of the particle $y$ and the time $t$ is

$$y = -\frac{1}{c} \ln\left(\cos fct - \frac{v}{f} \sin fct\right) \qquad \text{(equation 14·64)}$$

where

$$c = \frac{0{\cdot}33}{d} \cdot \frac{\rho}{\rho_s} = \frac{0{\cdot}33}{0{\cdot}25 \times 2{\cdot}54 \times 2{\cdot}5} = 0{\cdot}204 \qquad \text{(equation 14.50)}$$

$$f = \sqrt{\frac{d(\rho_s - \rho) \cdot g}{0{\cdot}33\rho}} = \sqrt{\frac{0{\cdot}25 \times 2{\cdot}54 \times 1{\cdot}5 \times 981}{0{\cdot}33 \times 1}} = 53{\cdot}5 \qquad \text{(equation 14.62)}$$

and

$$v = -16 \text{ f.p.s.} = -487 \text{ cm/sec.}$$

Thus $$y = -\frac{1}{0\cdot204}\ln\left(\cos 53\cdot5 \times 0\cdot204\, t + \frac{487}{53\cdot5}\sin 53\cdot5 \times 0\cdot204\, t\right)$$

$$= -4\cdot9 \ln(\cos 10\cdot8\, t + 9\cdot13 \sin 10\cdot8\, t)$$

$\therefore$ $$\dot{y} = -4\cdot9\,\frac{-10\cdot8 \sin 10\cdot8\, t + 9\cdot13 \times 10\cdot8 \cos 10\cdot8\, t}{\cos 10\cdot8\, t + 9\cdot13 \sin 10\cdot8\, t}$$

$$= -\frac{53\cdot2\,(9\cdot13 \cos 10\cdot8\, t - \sin 10\cdot8\, t)}{\cos 10\cdot8\, t + 9\cdot13 \sin 10\cdot8\, t}$$

The time taken for the velocity of the particle relative to the fluid to fall from 16 f.p.s. to 0·259 f.p.s. is given by—

$$-7\cdot9 = -\frac{53\cdot2\,(9\cdot13 \cos 10\cdot8\, t - \sin 10\cdot8\, t)}{\cos 10\cdot8\, t + 9\cdot13 \sin 10\cdot8\, t}$$

i.e. $$\cos 10\cdot8\, t + 9\cdot13 \sin 10\cdot8\, t = -6\cdot75 \sin 10\cdot8\, t + 61\cdot8 \cos 10\cdot8\, t$$

i.e. $$60\cdot8 \cos 10\cdot8\, t = 15\cdot28 \sin 10\cdot8\, t$$

$\therefore$ $$\sin 10\cdot8\, t = 3\cdot97 \cos 10\cdot8\, t$$

squaring, $$1 - \cos^2 10\cdot8\, t = 15\cdot7 \cos^2 10\cdot8\, t$$

$\therefore$ $$\cos 10\cdot8\, t = 0\cdot243 \qquad \ldots.(14\cdot67)$$

$\therefore$ $$\sin 10\cdot8\, t = \sqrt{1 - 0\cdot243^2} = 0\cdot97.$$

The distance moved by the particle relative to the fluid during this period is therefore given by

$$y = -4\cdot9 \ln(0\cdot243 + 9\cdot13 \times 0\cdot97)$$

$$= -4\cdot9 \ln 9\cdot07$$

$$= -10\cdot8 \text{ cm.}$$

If equation 14.64 were applied for a relative velocity down to zero, the time taken for the particle to come to rest would be given by

$$9\cdot13 \cos 10\cdot8\, t = \sin 10\cdot8\, t$$

squaring, $$1 - \cos^2 10\cdot8\, t = 83\cdot5 \cos^2 10\cdot8\, t$$

$\therefore$ $$\cos 10\cdot8\, t = 0\cdot109$$

and $$\sin 10\cdot8\, t = \sqrt{1 - 0\cdot109^2} = 0\cdot992$$

The corresponding distance the particle moves relative to the fluid is then given by—

$$y = -4\cdot9 \ln(0\cdot109 + 9\cdot13 \times 0\cdot992)$$

$$= -4\cdot9 \ln 9\cdot19$$

$$= -10\cdot9 \text{ cm}$$

i.e. the particle moves only a very small distance with a velocity of less than 0·259 f.p.s.

If form drag were neglected for all velocities less than 0·259 f.p.s., the distance moved by the particle would be given by equation 14.41.

$$y = \frac{b}{a}t + \frac{v}{a} - \frac{b}{a^2} + \left(\frac{b}{a^2} - \frac{v}{a}\right) \cdot e^{-at} \qquad \ldots.(14.41)$$

and

$$\dot{y} = \frac{b}{a} - \left(\frac{b}{a} - v\right) e^{-at}$$

where

$$a = 18\frac{\mu}{d^2\rho_s} = \frac{18 \times 0{\cdot}01}{\frac{1}{16} \times 2{\cdot}54^2 \times 2{\cdot}5} = 0{\cdot}1785, \qquad \text{(equation 14.39)}$$

$$b = \left(1 - \frac{\rho}{\rho_s}\right) \cdot g = 0{\cdot}6 \times 981 = 589, \qquad \text{(equation 14.40)}$$

$$b/a = 3280,$$

$$v = -7{\cdot}9 \text{ cm/sec.}$$

Thus

$$y = 3280\,t - \left(\frac{7{\cdot}9}{0{\cdot}1785} + \frac{3280}{0{\cdot}1785}\right).(1 - e^{-at})$$

$$= 3280\,t - \frac{3287{\cdot}9}{0{\cdot}1785}(1 - e^{-0{\cdot}1785\,t})$$

$$\therefore \quad y = 3280 - 3287{\cdot}9e^{-0{\cdot}1785\,t}$$

When the particle comes to rest in the fluid, $\dot{y} = 0$ and

$$e^{-0{\cdot}1785\,t} = \frac{3280}{3287{\cdot}9}$$

$$\therefore \quad t = 0{\cdot}013483 \text{ secs.}$$

The corresponding distance moved by the particle is given by

$$y = 3280 \times 0{\cdot}013483 - \frac{3287{\cdot}9}{0{\cdot}1785}\left(1 - \frac{3280}{3287{\cdot}9}\right)$$

$$= 44{\cdot}225 - 44{\cdot}258$$

$$= -0{\cdot}033 \text{ cm.}$$

Thus whether the resistance force is calculated by equation 14.8 or equation 14.12, the particle moves a negligible distance with a velocity relative to the fluid of less than 0·259 f.p.s. Further, the time is also negligible and thus the fluid also has moved through only a very small distance.

It can therefore be taken that the particle moves through 10·9 cm before it comes to rest in the fluid. The time taken for the particle to move this distance is given by equation 14.67, on the assumption that the drag force corresponds to that given by equation 14.12. The time is therefore given by

$$\cos 10{\cdot}8\,t = 0{\cdot}243 \qquad \text{(equation 14.67)}$$

$$\therefore \quad 10{\cdot}8\,t = 1{\cdot}325$$

$$t = 0{\cdot}123 \text{ secs.}$$

The distance travelled by the fluid in this time $= 30{\cdot}48 \times 4 \times 0{\cdot}123$

$$= 15{\cdot}0 \text{ cm.}$$

Thus the total distance moved by the particle relative to the walls of the plant

$$= 10{\cdot}9 + 15{\cdot}0 = 25{\cdot}9 \text{ cm}$$

$$= \underline{10{\cdot}2 \text{ inches.}}$$

## CHAPTER 15

# *Sedimentation, Fluidisation and Conveying*

IN the sedimentation of a concentrated suspension, particles move downwards under the action of gravity, and an equal volume of fluid is displaced. In fluidisation, a gas or liquid is passed upwards through a bed of particles sufficiently rapidly for each particle to be independently supported; the velocity of the fluid is, in general, less than the terminal falling velocity of the particles. At higher velocities, the particles are carried upwards in the fluid so that both solid and fluid move relative to the walls of the container. Whereas sedimentation and fluidisation involve vertical motion of the fluid, transport of particles may be carried out horizontally, vertically, or in any intermediate direction.

## SEDIMENTATION

When the concentration of a suspension is low, the distances between adjacent particles are large compared with the size of the particles and the effects of mutual interference can be disregarded. The rate of settling of a constituent particle under these conditions ("free settling") has been calculated in the previous chapter. At high concentrations, conditions within the suspension are considerably modified, particularly in that the upward velocity of the fluid displaced by the settling particles is much greater and the flow pattern is appreciably altered. The process is then known as "hindered settling" and is commonly encountered industrially in the concentration of suspensions by sedimentation in a thickener.

The sedimentation of metallurgical slimes has been studied by COE and CLEVENGER(11), who concluded that a concentrated suspension may settle in one of two different ways. In the first, after an initial brief acceleration period, the interface between the clear liquid and the suspension moves downwards at a constant rate and a layer of sediment builds up at the bottom of the container. When this interface approaches the layer of sediment, its rate of fall decreases until the "critical settling point" is reached when a direct interface is formed between the sediment and the clear liquid. Further sedimentation then results solely from a consolidation of the sediment, with liquid being forced upwards around the solids which are then forming a loose bed with the particles in contact with one another. Since the flow area is gradually being reduced, the rate progressively diminishes. In Fig. 15.1*a*, a stage in the sedimentation process is illustrated. *A* is clear liquid, *B* is suspension of the original concentration, *C* is a layer through which the concentration gradually increases, and *D* is sediment. The sedimentation rate remains constant until the upper interface corresponds with the top of zone *C* and it then falls until the critical settling point is reached when both zones *B* and *C* will have disappeared. A second and rather less common mode of sedimentation (Fig. 15.1*b*) is obtained when the range of particle size is very great. The sedimentation rate progressively decreases throughout the whole operation because there is no zone of constant composition, and zone *C* extends from the top interface to the layer of sediment.

The main reasons for the modification of the settling rate of particles in a concentrated suspension are as follows:

(*a*) The large particles are settling relative to a suspension of smaller ones so that the effective density and viscosity of the fluid are increased.

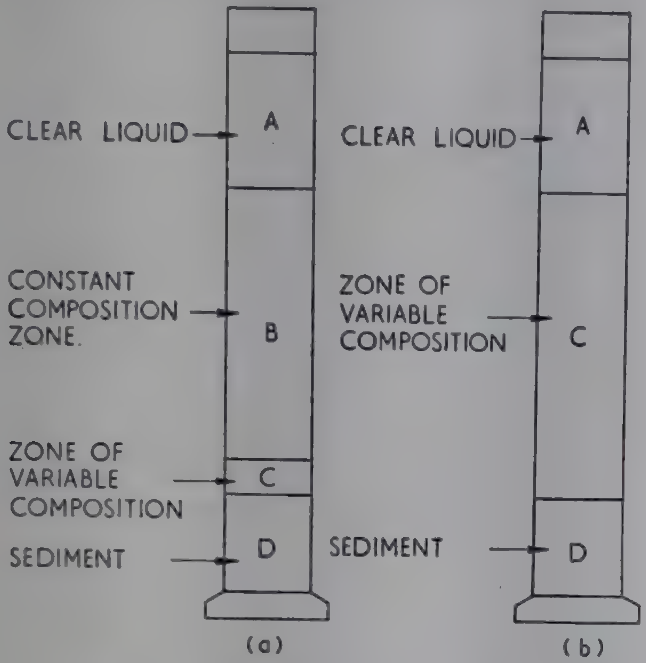

CLEAR LIQUID
A
CONSTANT COMPOSITION ZONE.
B
ZONE OF VARIABLE COMPOSITION
C
SEDIMENT
D
(a)
CLEAR LIQUID
A
ZONE OF VARIABLE COMPOSITION
C
SEDIMENT
D
(b)

Fig. 15.1. Sedimentation of concentrated suspensions
(a) Type 1 settling (b) Type 2 settling

(*b*) The upward velocity of the fluid displaced during settling is appreciable in a concentrated suspension and the apparent settling velocity is less than the actual velocity relative to the fluid.

(*c*) The velocity gradients in the fluid close to the particles are increased as a result of the change in the area and shape of the flow spaces.

(*d*) The smaller particles tend to be dragged downwards by the motion of the large particles and therefore accelerated.

(*e*) Because the particles are closer together in a concentrated suspension, flocculation is more marked in an ionised solvent and the effective size of the small particles is increased.

If the range of particle size is not more than about 6:1, a concentrated suspension settles with a sharp interface and all the particles fall at the same velocity. This is in contrast with the behaviour of a dilute suspension, for which the rates of settling of the particles can be calculated by the methods given in Chapter 14, and where the settling velocity is greater for the large particles.

The overall result is that in a concentrated suspension the large particles are retarded and the small ones accelerated.

A number of attempts have been made to predict the apparent settling velocity of a concentrated suspension. In 1926, ROBINSON(14) suggested a modification of STOKES' Law and used the density ($\rho_c$) and viscosity ($\mu_c$) of the suspension in place of the properties of the fluid.

Thus
$$u_c = \frac{K'' d^2(\rho_s - \rho_c)g}{\mu_c} \qquad \ldots.(15.1)$$

where $K''$ is a constant.

The effective buoyancy force is readily calculated since

$$\rho_s - \rho_c = \rho_s - \{\rho_s(1-e) + \rho e\} = e(\rho_s - \rho) \qquad \ldots.(15.2)$$

where $e$ is the voidage of the suspension.

ROBINSON determined the viscosity of the suspension $\mu_c$ experimentally, but it may be obtained approximately from the following formula of EINSTEIN(7)

$$\mu_c = \mu(1 + k''C) \qquad \ldots.(15.3)$$

where $k''$ is a constant for a given shape of particle ($\frac{5}{2}$ for spheres),
$C$ is the volumetric concentration of particles, and
$\mu$ is the viscosity of the fluid.

This equation holds for values of $C$ up to 0·02. For more concentrated suspensions, VAND(40) gives the equation

$$\mu_c = \mu e^{\frac{k''C}{1-qC}} \qquad \ldots.(15.4)$$

in which $q$ is a second constant, equal to $\frac{39}{64}$ for spheres.

STEINOUR(25), who studied the sedimentation of small uniform particles, adopted a similar approach, using the viscosity of the fluid, the density of the suspension and a function of the voidage of the suspension to take account of the character of the flow spaces, and obtained an expression for the velocity of the particle relative to the fluid $u_p$ as follows,

$$u_p = \frac{d^2(\rho_s - \rho_c)g}{18\mu} \cdot f(e) \qquad \ldots.(15.5)$$

Since the fraction of the area available for flow of the displaced fluid is $e$, its upward velocity is $u_c \frac{1-e}{e}$ so that

$$u_p = u_c + u_c \frac{1-e}{e} = \frac{u_c}{e}. \qquad \ldots.(15.6)$$

From his experiments on the sedimentation of tapioca in oil STEINOUR has found

$$f(e) = 10^{-1\cdot 82(1-e)} \qquad \ldots.(15.7)$$

Substituting in equation 15.5, from equations 15.2, 15.6 and 15.7,

$$u_c = \frac{e^2 d^2(\rho_s - \rho)g}{18\mu} \, . \, 10^{-1\cdot 82(1-e)} \qquad \ldots.(15.8)$$

HAWKSLEY(50) also used a similar method and gave

$$u_p = \frac{u_c}{e} = \frac{d^2(\rho_s - \rho_c)g}{18\mu_c} \qquad \ldots.(15.9)$$

In each of the above cases, it is assumed that the effective gravitational force acting on the particles depends on the density of the suspension rather than that of the fluid. Whereas this cannot be true if all the particles are settling at the same velocity, because each particle then displaces its own volume of fluid—not of suspension—it is approximately correct for a large particle settling in a fine suspension because the suspension then behaves in a similar manner to a homogeneous fluid. Again, the use of an effective viscosity is valid only for a large particle settling in a fine suspension. For the sedimentation of uniform particles the increased drag is attributable to a steepening of the velocity gradients rather than to a change in viscosity.

The rate of sedimentation of a suspension of fine particles is difficult to predict because of the large number of factors involved. Thus, for instance, the presence of an ionised solute in the liquid and the nature of the surface of the particles will affect the degree of flocculation and hence the mean size and density of the flocs. The flocculation of a suspension is usually completed quite rapidly so that it is not possible to detect an increase in the sedimentation rate in the early stages after the formation of the suspension. A further factor influencing the sedimentation rate is the degree of agitation of the suspension. Gentle stirring may produce accelerated settling if the suspension behaves as a non-Newtonian fluid in which the apparent viscosity is a function of the rate of shear. The change in apparent viscosity can probably be attributed to the re-orientation of the particles. The effect of stirring is, however, most marked on the consolidation of the final sediment, in which "bridge formation" by the particles can be prevented by gentle stirring. During these final stages of consolidation of the sediment, liquid is being squeezed out through a bed of particles which are gradually becoming more tightly packed.

A number of empirical equations have been obtained for the rate of sedimentation of suspensions, as a result of tests carried out in vertical tubes. For a given solid and liquid, the chief factors which affect the process are the height of the suspension, the diameter of the containing vessel, and the volumetric concentration.

***Height of Suspension.*** The height of suspension does not generally affect either the rate of sedimentation or the consistency of the sediment ultimately obtained. If, however, the position of the sludge line is plotted as a function of time for two different initial heights of slurry, curves of the form shown in Fig. 15.2 are obtained in which the ratio OA′ : OA″ is everywhere constant. Thus if the curve is obtained for any one initial height, the curves can be drawn for any other height.

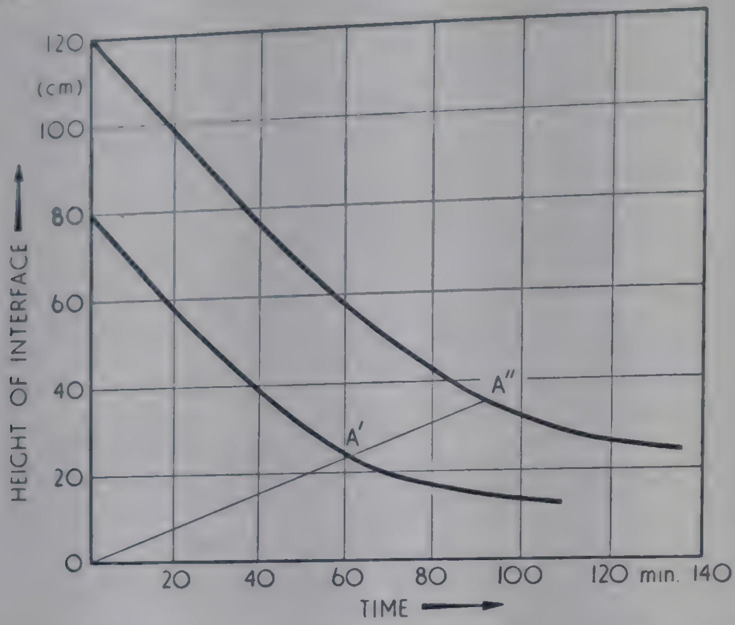


Fig. 15.2. Effect of height on sedimentation of 3% (volume) suspension of calcium carbonate

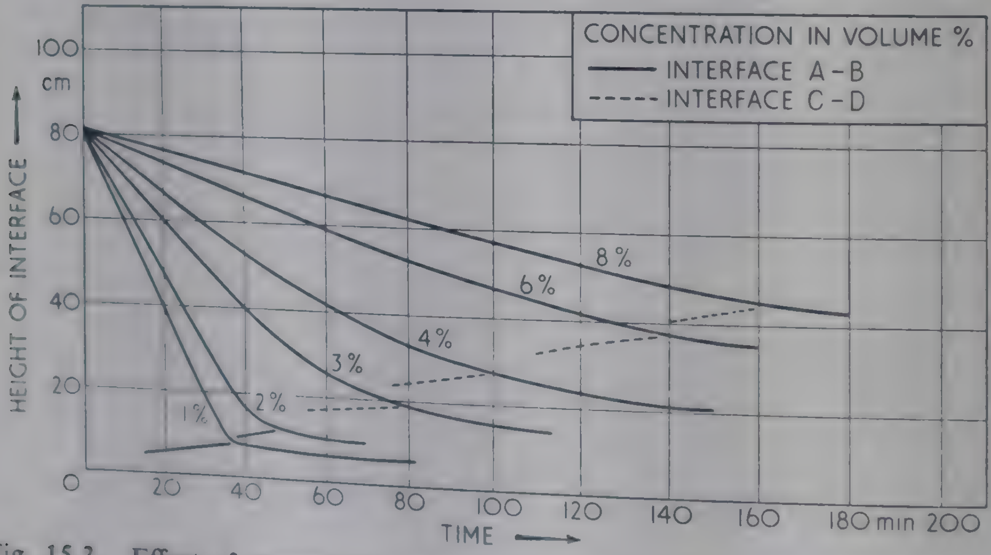


Fig. 15.3. Effect of concentration on sedimentation of calcium carbonate suspensions

***Diameter of Vessel.*** If the ratio of the diameter of the vessel to the diameter of the particle is greater than about 100, the walls of the container appear to have no effect on the rate of sedimentation. For smaller values, the sedimentation rate may be reduced because of the retarding influence of the walls.

***Concentration of Suspension.*** As already indicated, the higher the concentration, the lower is the rate of fall of the sludge line because the greater is the upward velocity of the displaced fluid and the steeper are the velocity gradients in the fluid. Typical curves for the sedimentation of a suspension of precipitated calcium carbonates in water are shown in Fig. 15.3, and in Fig. 15.4 the mass rate of sedimentation (gm/cm²-hr) is plotted against the concentration. This curve shows a maximum corresponding to a volumetric concentration of about 2%. EGOLF and McCABE(18), WORK and KOHLER(23), and others have given empirical expressions for the rate of sedimentation at the various stages, but these are generally applicable over a narrow range of conditions and involve constants which need to be determined experimentally for each suspension.

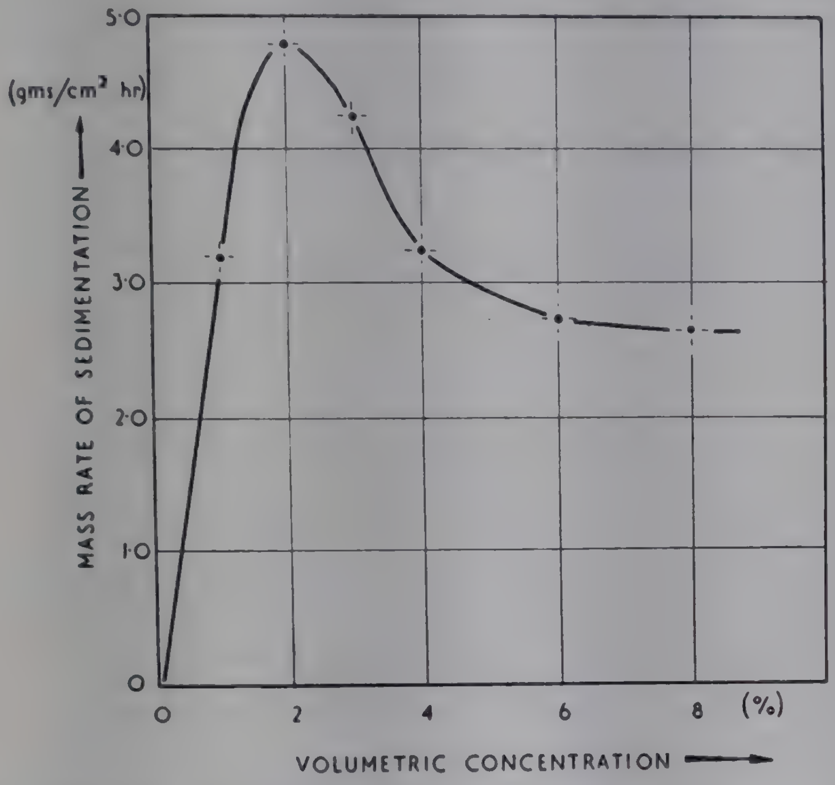


Fig. 15.4. Effect of concentration on mass rate of sedimentation of calcium carbonate

The final consolidation of the sediment is the slowest part of the process because the displaced fluid has to flow through the small spaces between the particles. As consolidation occurs, the rate falls off because the resistance to the flow of liquid progressively increases. The porosity of the sediment is smallest at the bottom because the compressive force due to the weight of particles is greatest and because the lower portion was formed at an earlier stage in the sedimentation process. The rate of sedimentation during this period is given approximately by the expression,

$$-\frac{\mathrm{d}H}{\mathrm{d}t} = i(H - H_\infty) \qquad \ldots.(15.10)$$

where $H$ is the height of the sludge line at time $t$,

$H_\infty$ is the final height of the sediment, and

$i$ is constant for a given suspension.

The time taken for the sludge line to fall from a height $H_c$, corresponding to the critical settling point, to a height $H$ is given by

$$-it = \ln (H - H_\infty) - \ln (H_c - H_\infty) \qquad \text{....(15.11)}$$

Thus, if $\ln (H - H_\infty)$ is plotted against $t$, a straight line of slope $-i$ is obtained.

The value of $H_\infty$ is determined largely by the surface film of liquid adhering to the particles.

**Coarse Suspensions**

In a recent paper by RICHARDSON and ZAKI(83), the sedimentation of uniform particles (>100 microns), sufficiently large for anomalous viscosity effects and flocculation to be negligible, is discussed, and experimental work on sedimentation and fluidisation is used to establish the effects of concentration and of the walls of the container on sedimentation rates.

The drag $R_t$ per unit projected area for a spherical particle settling at its terminal falling velocity $u_0$ is a function of the density and viscosity of the fluid, the diameter of the particle, the terminal falling velocity, and the ratio of the diameter of the particle to that of the containing vessel,

i.e.
$$R_t = f\left(\rho, \mu, u_0, d, \frac{d}{D}\right) \qquad \text{....(15.12)}$$

The corresponding value of $R_t$ for the sedimentation of a particle in a suspension will be the same, since in each case $R_t \frac{\pi}{4} d^2$ is equal to the weight of the particle in the fluid. In this case, however, the drag will be produced as a result of a smaller relative velocity $u_p$ and will, in addition, be a function of the voidage $e$ of the suspension as this will affect the flow pattern and determine the free area available for the flow of the displaced fluid, so that

$$R_t = f\left(\rho, \mu, u_p, d, e, \frac{d}{D}\right) \qquad \text{....(15.13)}$$

Re-arranging these two equations to give $u_0$ and $u_p$ explicitly, and dividing,

$$\frac{u_p}{u_0} = f\left(R_t, \rho, \mu, d, e, \frac{d}{D}\right) \qquad \text{....(15.14)}$$

Since the left hand side of equation 15.14 is dimensionless the right hand side must also be. The only dimensionless combinations of the variables $R_t$, $\rho$, $\mu$ and $d$ involve the group $\frac{R_t d^2 \rho}{\mu^2}$, and therefore

$$\frac{u_p}{u_0} = f\left(\frac{R_t d^2 \rho}{\mu^2}, e, \frac{d}{D}\right) \qquad \text{....(15.15)}$$

Now $\frac{R_t d^2 \rho}{\mu^2} = \frac{R_t}{\rho u_0^2} \cdot \frac{u_0^2 d^2 \rho^2}{\mu^2}$, and since $\frac{R_t}{\rho u_0^2}$ is a unique function of $\frac{u_0 d \rho}{\mu}$ (= $Re.'$) for a spherical particle (see page 483),

$$\frac{u_p}{u_0} = f\left(Re.', e, \frac{d}{D}\right) \qquad \text{....(15.16)}$$

The measured sedimentation velocity $u_c$ is given in terms of the velocity of the particles relative to the fluid by equation 15.6

$$u_c = eu_p \qquad \ldots.(15.6)$$

Thus
$$\frac{u_c}{u_0} = f\left(Re.', e, \frac{d}{D}\right) \qquad \ldots.(15.17)$$

At low velocities ($Re.' < 0{\cdot}2$) where the drag force is attributable entirely to skin friction, from equation 14.1;

$$R_t \frac{\pi}{4} d^2 = 3\pi\mu d u_0 \qquad \ldots.(15.18)$$

and $R_t$ is independent of the density of the fluid.

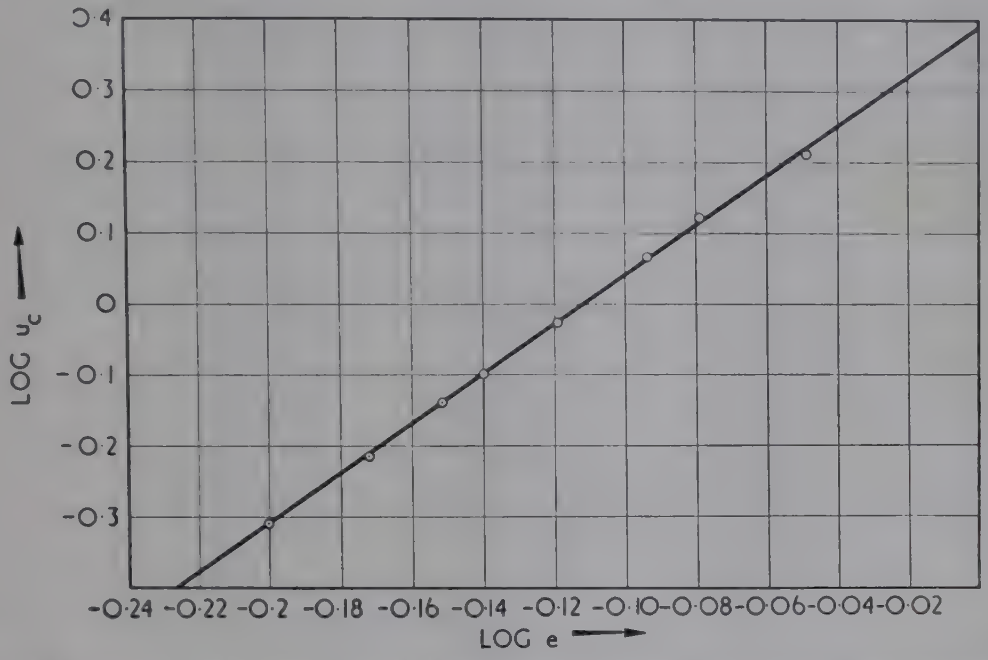


Fig. 15.5. **Relation between rate of sedimentation and voidage of suspensions of 1 mm glass balls in bromoform**

Again when the Reynolds Group $Re.'$ exceeds 500, skin friction is negligible and

$$R_t \propto \rho u_0^2 \qquad \ldots.(15.19)$$

$R_t$ is then independent of $\mu$ and $d$.

Neither $R_t$, $\mu$ and $d$, nor $R_t$ and $\rho$ can be arranged in the form of a dimensionless group, and therefore the ratio $\frac{u_c}{u_0}$ is independent of $Re.'$, when $Re.'$ is less than 0·2 or greater than 500.

i.e.
$$\frac{u_c}{u_0} = f\left(e, \frac{d}{D}\right) \qquad \ldots.(15.20)$$

The results for the sedimentation of uniform spheres and for the fluidisation experiments referred to on page 527, were plotted as $\log u_c$ versus $\log e$ (e.g. as in Fig. 15.5), for each suspension and a linear relationship was obtained so that

$$\log u_c = n \log e + \log u_i \qquad \ldots.(15.21)$$

where $\log u_i$ is the intercept corresponding to infinite dilution ($e = 1$).

Thus

$$\frac{u_c}{u_i} = e^n \qquad \ldots.(15.22)$$

$u_i$ corresponds to the falling velocity of the suspension in a tube of diameter $D$ at infinite dilution and therefore

$$\frac{u_i}{u_0} = f\left(\frac{d}{D}\right) \text{ only} \qquad \ldots.(15.23)$$

Thus from equations 15.22, 15.23 and 15.17,

$$e^n = f\left(Re.', e, \frac{d}{D}\right)$$

and since $n$ is independent of $e$,

$$n = f\left(Re.', \frac{d}{D}\right) \qquad \ldots.(15.24)$$

For $Re.' < 0{\cdot}2$, and for $Re.' > 500$, from equation 15.20,

$$n = f\left(\frac{d}{D}\right) \text{ only} \qquad \ldots.(15.25)$$

The experimental values of $n$ have been plotted against $\dfrac{d}{D}$ in Fig. 15.6 and are correlated by a family of straight lines. As expected, a single curve represents the data for all values of $Re.'$ less than 0·2, and another single curve for all values of $Re.'$ greater than 500. At intermediate values, $n$ is a function of $Re.'$ From an analysis of the curves

For $0 < Re.' < 0{\cdot}2$

$$n = 4{\cdot}6 + 20\frac{d}{D} \qquad \ldots.(15.26)$$

$0{\cdot}2 < Re.' < 1$

$$n = \left(4{\cdot}4 + 18\frac{d}{D}\right) Re.'^{-0{\cdot}03} \qquad \ldots.(15.27)$$

$1 < Re.' < 200$

$$n = \left(4{\cdot}4 + 18\frac{d}{D}\right) Re.'^{-0{\cdot}1} \qquad \ldots.(15.28)$$

$200 < Re.' < 500$

$$n = 4{\cdot}4\, Re.'^{-0.1} \qquad \ldots.(15.29)$$

$Re.' > 500$

$$n = 2{\cdot}4 \qquad \ldots.(15.30)$$

It was found that $u_0$ and $u_i$ were equal for the sedimentation experiments, and for the fluidisation tests

$$\log u_0 = \log u_i + \frac{d}{D} \qquad \ldots.(15.31)$$

The calculation of the sedimentation rate in terms of the free falling velocity therefore involves obtaining the index *n* by inserting the values of $\frac{d}{D}$ and *Re.'* in the appropriate equation (15.26–15.30 inclusive) and substituting for $u_i$ in terms of $u_0$. The sedimentation velocity $u_c$ is then obtained from equation 15.22. For non-spherical particles, the index *n*, as calculated above, must be multiplied by a factor of $1{\cdot}1k'^{0{\cdot}165}$, where $k'$ is the shape factor as defined on page 494: this has been established only at values of *Re.'* greater than 500.

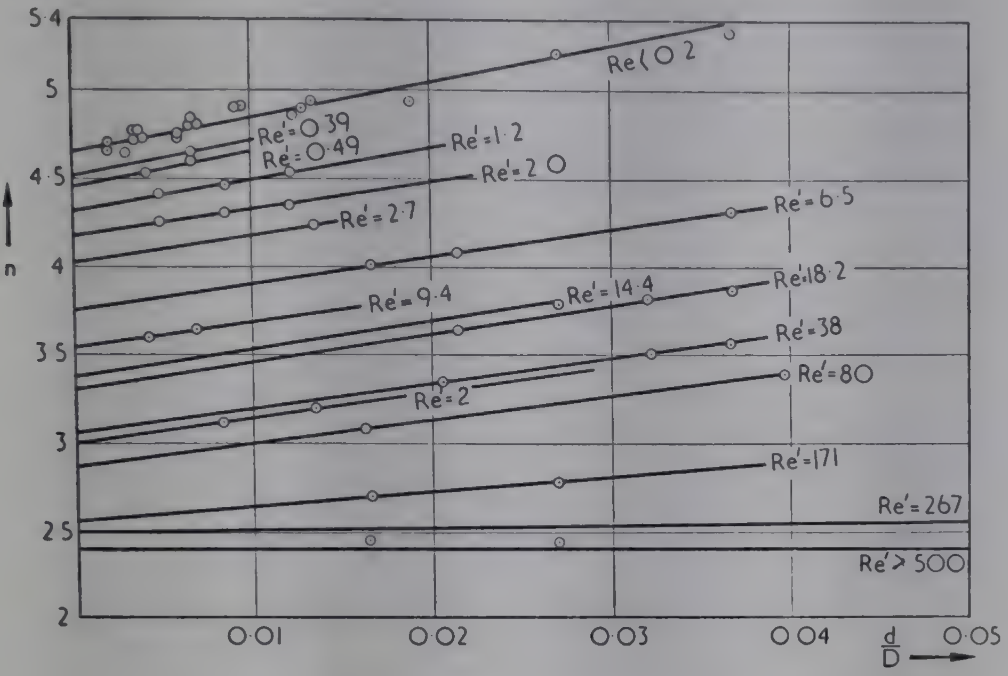


Fig. 15,6. *n* for various values of *Re.'* and $\frac{d}{D}$

## The Thickener

The thickener is the industrial plant in which the concentration of a suspension is increased by sedimentation, with the formation of a clear liquid. In most cases, the concentration of the suspension is high and hindered settling takes place. Thickeners may operate as batch or continuous units, and consist of relatively shallow tanks from which the clear liquid is taken off at the top and the thickened liquor at the bottom.

In order to obtain the largest possible throughput from a thickener of given size, the rate of sedimentation should be as high as possible. In many cases, the rate may be artificially increased by the addition of small quantities of an electrolyte, which causes precipitation of colloidal particles and the formation of flocs. The suspension is also frequently heated because this lowers the viscosity of the liquid, and encourages the larger particles in the suspension to grow in size at the expense of the more soluble small particles. Further the thickener frequently incorporates a slow stirrer, which causes a reduction in the apparent viscosity of the suspension and also aids in the consolidation of the sediment.

The batch thickener usually consists of a cylindrical tank with a conical bottom. After sedimentation has proceeded for an adequate time, the thickened liquor is withdrawn from the bottom and the clear liquid is taken off through an adjustable offtake pipe from the upper part of the tank. The conditions prevailing in the batch thickener are similar to those in the ordinary laboratory sedimentation tube, and during the initial stages there will generally be a zone in which the concentration of the suspension is the same as that in the feed.

The continuous thickener, such as the Dorr thickener (Fig. 15.7), consists of a large diameter shallow tank with a flat bottom. The liquor is fed in at the centre, at a depth of from one to three feet below the surface of the liquid, with as little disturbance as possible. The thickened liquor is continuously removed through an outlet at the bottom, and any solids which are deposited on the floor of the tank are directed towards the outlet by means of a slowly rotating rake mechanism incorporating scrapers. The rake is hinged so that the arms fold up automatically if the torque exceeds a certain value; this prevents it from being damaged if it is overloaded. Because of the action of the rake, a greater degree of thickening is obtained than with the batch plant. The clarified liquid is continuously removed from an overflow which runs round the whole of the upper edge of the tank. The solids are therefore moving continuously downwards, and then inwards towards the thickened liquor outlet; the liquid is moving upwards and radially outwards (Fig. 15.8). In general, there will be no region of constant composition in the continuous thickener.

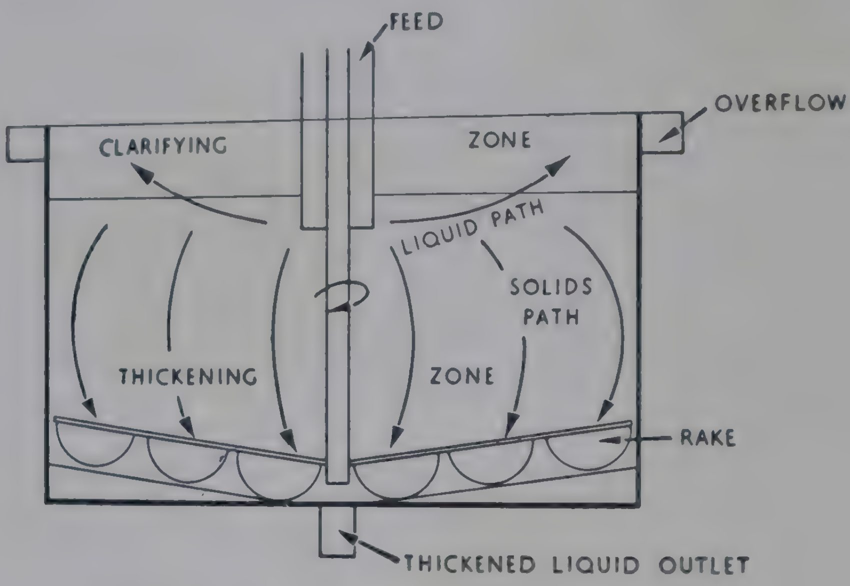


Fig. 15.8. Flow in continuous thickener

The thickener has a two-fold function. First it must produce a clarified liquid, and therefore the upward velocity of the liquid must, at all times, be less than the settling velocity of the particles. Thus, for a given throughput, the clarifying capacity is determined by the diameter of the tank. Secondly, the thickener is required to produce a given degree of thickening of the suspension. This is controlled by the time of residence of the particles in the tank, and hence by the depth below the feed inlet.

The satisfactory operation of the thickener as a clarifier depends upon the

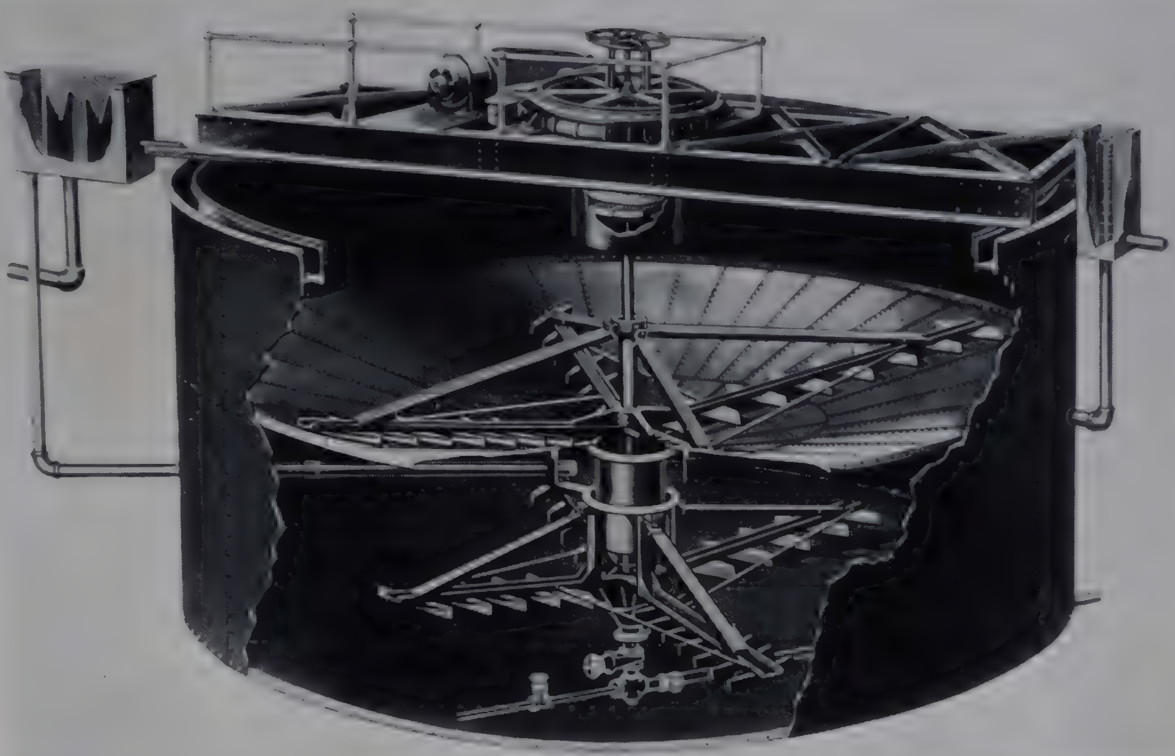


Fig. 15.7. Dorr thickener—two trays

existence of a zone of negligible solid content towards the top. In this zone conditions approach those under which free settling takes place, and the rate of sedimentation of any particles which have been carried to this height is therefore sufficient for them to settle against the upward current of liquid. If this upper zone is too shallow, some of the smaller particles may escape in the liquid overflow. The volumetric rate of flow of liquid upwards through the clarification zone is equal to the difference between the rate of feed of liquid in the slurry and the rate of removal in the underflow. Thus the required concentration of solids in the underflow, as well as the throughput, determines the conditions in the clarification zone.

If $U$ is the mass ratio of liquid to solid in the feed and $V$ is the corresponding value required in the underflow, the mass rate of discharge of liquid in the overflow, per unit rate of feed of solids, is $U - V$. Then, if $W$ is the mass rate of feed of solids, the upward velocity of liquid in the clarification zone is $\frac{(U-V)\,.\,W}{A\,.\,\rho}$, where $A$ is the cross-sectional area of the tank and $\rho$ is the density of the liquid. This velocity must not exceed the rate of sedimentation of the slurry $u_c$ and therefore the required area for successful operation is given by,

$$A = \frac{(U-V)\,.\,W}{u_c\,.\,\rho} \qquad \ldots.(15.32)$$

The values of $A$ should be calculated for the whole range of concentrations present in the thickener, and the design should then be based on the maximum value so obtained.

The concentration of solids in the underflow will be a function of the depth of the tank below the feed point and the time of residence of the solids in the thickener. In most cases, the time of compression of the sediment will be large compared with the time taken for the critical settling conditions to be reached for any portion of the slurry. The time required to concentrate the sediment after it has reached the critical condition can be determined approximately by allowing a sample of the slurry at its critical composition to settle in a vertical glass tube and measuring the time taken for the interface between the sediment and the clear liquid to fall to such a level that the concentration is that required in the underflow from the thickener. The use of data so obtained assumes that the average concentration in the sediment in the laboratory test is the same as that which would be obtained in the thickener after the same time. This is not quite so because, in the thickener, the various parts of the sediment have been under compression for different times. Further, it assumes that the time taken for the sediment to increase in concentration by a given amount is independent of its depth.

An approximate value for the depth of the thickening zone is then found by adding the volume of the liquid in the sediment to the corresponding volume of solid and dividing by the area, which has already been calculated in order to determine the clarifying capacity of the thickener. The required depth of the thickening region is thus

$$\left\{\frac{Wt_r}{A\rho_s} + W\frac{t_r}{A\rho}X\right\} = \frac{Wt_r}{A\rho_s}\left(1 + \frac{\rho_s}{\rho}X\right) \qquad \ldots.(15.33)$$

where $t_r$ is the required time of retention of the solids, as determined experimentally,

$W$ is the mass rate of feed of solids to the thickener,

$X$ is the average value of the mass ratio of liquid to solids in the thickening portion,

and $\rho$ and $\rho_s$ are the densities of the liquid and solid respectively.

This method of design is only approximate and therefore, in a large tank, about three feet should be added to the calculated depth as a safety margin and to allow for the depth required for the suspension to reach the critical concentration. In addition, the bottom of the tanks may be slightly pitched to assist the flow of material towards the thickened liquor outlet. The diameter of the tank is usually large compared with its depth and therefore a large ground area is required. Space is often saved by arranging a number of thickeners vertically above one another. They operate as separate units but a common central shaft is utilized to drive the rakes and stirrers.

Thickeners may be anything from a few feet to several hundred feet in diameter. The small ones are made of wood or metal and the rakes rotate at about 1 r.p.m. The very large thickeners generally consist of large concrete tanks and the stirrers and rakes are driven by means of traction motors which drive on a rail running round the whole circumference; the speed of rotation may be as low as 0·1 r.p.m.

## FLUIDISATION

If a fluid is passed downwards through a bed of solids, no relative movement between the particles takes place, unless the initial orientation is unstable. If the flow is streamline, the pressure drop across the bed will be directly proportional to the rate of flow but at higher rates it will rise more rapidly. The expressions given in Chapter 11 can be used for the calculation of the pressure drop under these conditions.

If the fluid passes upwards through the bed, the pressure drop will be the same as for downward flow at low rates, but when the frictional drag on the particles becomes equal to their apparent weight (actual weight less buoyancy), the particles become rearranged so that they offer less resistance to the flow of fluid and the bed starts to expand. This process continues as the velocity is increased, with the total frictional force remaining equal to the weight of the particles, until the bed has assumed the loosest stable form of packing. If the velocity is then increased still further, the individual particles separate from one another and become freely supported in the fluid and the bed is said to be fluidised. Further increase in the velocity causes the particles to separate still further from one another, and the pressure difference remains approximately equal to the weight per unit area of the bed.

Up to this stage the system behaves in a similar way whether the fluid is a liquid or gas, but at high fluid velocities when the expansion of the bed is large there is a fairly sharp distinction between the behaviour in the two cases. With a liquid, the bed continues to expand as the velocity is increased and it maintains its uniform character, with the amount of turbulence and the agitation of the particles increasing progressively. This type of fluidisation is known as "particulate fluidisation". With a gas, however, uniform fluidisation is obtained only at relatively low velocities and at high velocities two separate "phases" are formed; these are often referred to as the dense phase and the lean phase and a condition of "aggregative" fluidisation is produced. Gas containing a relatively small proportion of suspended solids bubbles through a high-density fluidised bed with the result that the system closely resembles a boiling liquid in appearance, with the lean phase corresponding to the vapour and the dense or continuous phase to the liquid. The bed is then often referred to as a "boiling bed", as opposed to a "quiescent bed" at low flow rates. Thus as the flow of the gas is increased, its velocity relative to the particles in the dense phase may not change appreciably, and it has been suggested(71) that the flow relative to the particle can as a result remain streamline even at very high rates of flow. If the rate of passage of gas is high, and if the bed is deep, coalescence of the bubbles takes place and eventually slugs of gas occupying the whole cross-section of the containing vessel are produced. These slugs of gas alternate with slugs of fluidised solids which are carried upwards and subsequently collapse causing the solids to fall back again. If the gas velocity is now increased so that it exceeds the terminal falling velocity of the particles, in the absence of channelling the slugs do not disintegrate but continue to rise indefinitely and transport of the solids takes place in the gas stream. At still higher velocities the solid becomes better dispersed in the gas and the mixture behaves similarly to a single phase system.

It has been found that the Froude Group $\frac{u_c^2}{gd}$ gives a criterion from which the type of fluidisation can be predicted.

Here $u_c$ is the velocity of flow, calculated over the whole cross-section of the bed, at which fluidisation takes place,

$d$ is the diameter of the particles, and

$g$ is the acceleration due to gravity.

At values less than unity, particulate fluidisation occurs but, at higher values, aggregative fluidisation takes place. Much lower values of the Froude Number are generally obtained with liquids because the velocity required to produce fluidisation is less.

Although the possibility of forming fluidised beds has been known for many years, the subject has remained of academic interest until the recent adoption of fluidised catalysts by the petroleum industry for the cracking of heavy hydrocarbons and for the synthesis of fuels from natural gas and from carbon monoxide and hydrogen. The fluidised bed in many ways behaves as a fluid of the same density as that of the solids and fluid combined. It transmits hydrostatic forces and solid objects float if their densities are lower than that of the bed. Intimate mixing takes place and, because of the high degree of turbulence, heat transfer within the bed is very rapid and therefore uniform temperatures are quickly attained throughout the system. The easy control of temperature is the feature which has led to the development of fluidised solids for catalytic cracking processes.

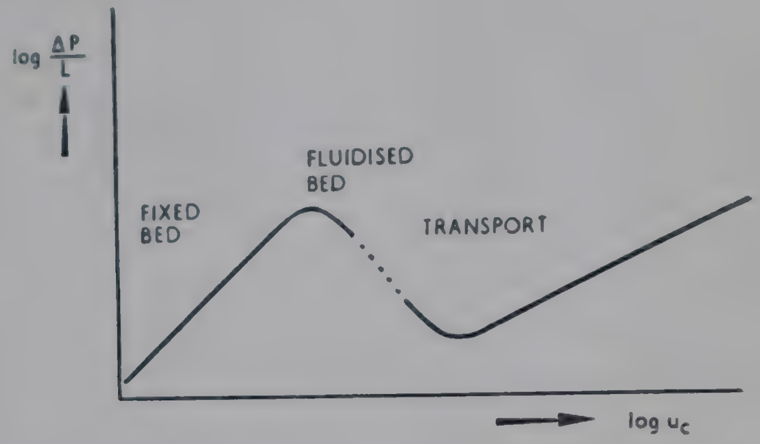


Fig. 15.9. Pressure gradient in bed as function of fluid velocity

### Effect of Fluid Velocity on Expansion of Bed and Pressure Gradient

The relation between the superficial velocity $u_c$ of the fluid (calculated over the whole cross-section of the containing vessel) and the pressure gradient is shown in Fig. 15.9. Here the flow is streamline and the curve is a straight line of slope unity. At the fluidising point, the pressure gradient begins to fall because the porosity of the bed increases; this continues until the velocity is high enough for transport of the material to take place; the pressure gradient then starts to increase again because the frictional drag of the fluid at the walls of the tube starts to become significant.

If the pressure across the whole bed instead of the pressure gradient is plotted against velocity, again using logarithmic coordinates (Fig. 15.10), a linear relation is again obtained up to the point where expansion of the bed takes place ($A$), but the slope of the curve then gradually diminishes as the bed expands. As the velocity is increased, the pressure drop passes through a maximum value ($B$) and then falls slightly and attains an approximately constant value independent of fluid velocity ($CD$). If the velocity of flow is reduced again, the bed contracts until it reaches the condition where the particles are just resting on one another ($E$); it then has the maximum stable porosity for a fixed bed of the particles in question. If the velocity is further decreased the bed then remains in this condition provided that it is not shaken. The pressure drop ($EF$) in this reformed fixed bed is then less than that originally obtained at the same velocity. If the velocity were now increased again, it would be expected that the new curve (*FE*) would be retraced and that the slope would suddenly change from 1 to 0 at the fluidising point, but this condition is difficult to produce because the bed tends to become consolidated again unless it is completely free from vibration. In the absence of channelling, the shape and size of the particles alone determines both the maximum porosity and the pressure drop at fluidisation for a bed of given dimensions.

The velocity corresponding to the fluidising point is readily calculated from the equations given in Chapter 11. The pressure drop through the bed is then equal to its apparent weight per unit area, and the porosity at the onset of fluidisation is the maximum which the fixed bed can attain. VAN HEERDEN(56) has suggested that the porosity term be eliminated from the above equations for the drop in pressure through a fixed bed by combining it with the numerical constant, because both the constant and the maximum porosity are functions solely of particle shape.

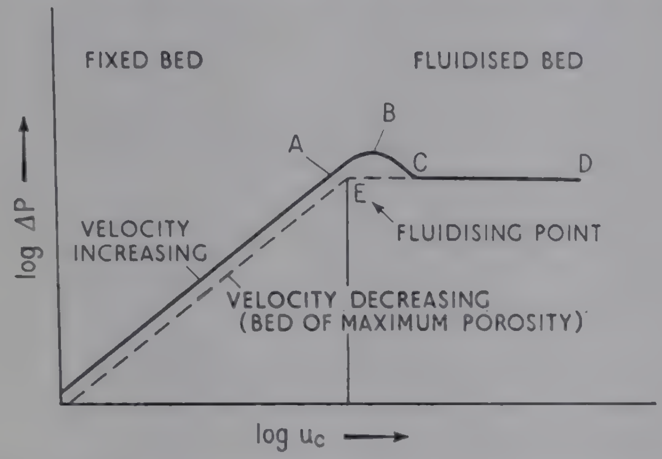


Fig. 15.10. **Pressure drop over fixed and fluidised beds**

In a fluidised bed, the total frictional force on the particles must be equal to the effective weight of the bed. Thus in a bed of unit cross-sectional area, depth $L$, and porosity $e$, we have

$$\text{Total Force} = \triangle P = (1 - e)(\rho_s - \rho) \,.\, L \,.\, g \qquad \ldots.(15.34)$$

where $\triangle P$ is the pressure drop across the bed, and
$g$ is the acceleration due to gravity.

This relation applies from the initial expansion of the bed until transport of solids takes place. There may be some discrepancy between the calculated and measured minimum velocities for fluidisation. This may be attributable to channelling, as a result of which the drag force acting on the bed is reduced, to the action of electrostatic forces in the case of gaseous fluidisation (these are particularly important in the case of sands), to agglomeration which is often considerable with small particles, or to friction between the fluid and the walls of the containing vessel: this last factor is of greatest importance with beds of small diameters. LEVA(47) has introduced a term $\dfrac{G_F - G_E}{G_F}$, a fluidisation efficiency, in which $G_F$ is the flow rate of fluid required to produce fluidisation and $G_E$ is the rate which will cause an initial expansion of the bed.

The minimum mass rate of flow which will produce fluidisation if flow conditions within the bed are streamline is obtained by substituting from equation 11.7 into equation 15.34 to give, for spherical particles,

$$\rho u = 0{\cdot}0055 \frac{e^3}{1-e} d^2 \frac{(\rho_3 - \rho)\rho g}{\mu} \qquad \ldots.(15.35)$$

Here $e$ refers to the voidage immediately before fluidisation occurs.

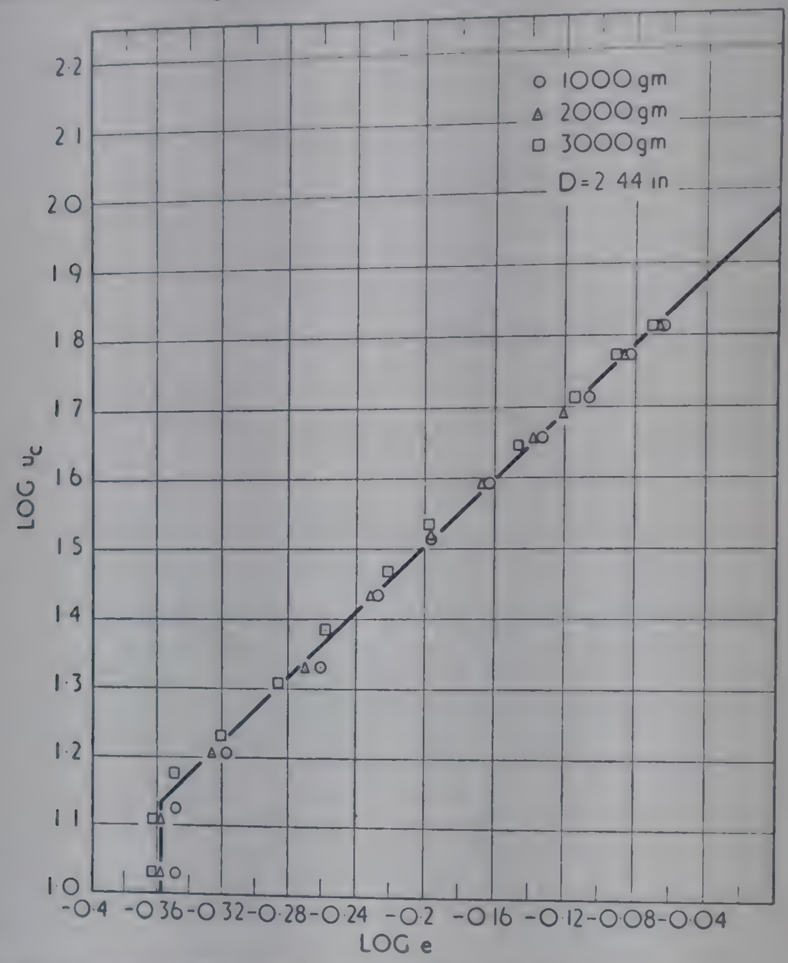


Fig. 15.11. Relation between fluid velocity ($u_c$) and voidage for fluidisation of ¼ in. steel spheres in water

For fluidisation with a liquid, if the voidage $e$ of the bed is plotted against the velocity of the fluid $u_c$ (using logarithmic co-ordinates) (Fig. 15.11), the curve can be divided approximately into two straight lines, joined by a short curve. At low velocities the voidage remains constant, and for the fluidised state there is a linear relation between log $u_c$ and log $e$ giving an intercept of log $u_i$ and a slope $n$, say. We can therefore write

$$\frac{u_c}{u_i} = e^n \qquad \ldots.(15.36)$$

$u_i$ is found to be very close to the terminal falling velocity of the particles, except when the ratio $\frac{d}{D}$ is comparatively large. Equation 15.36 gives a similar relationship between $u_c$ and $e$ to that obtained for sedimentation experiments (see page 517). For liquid fluidisation, the porosity of the bed can be then calculated as a function of the flow rate by the method already given on page 519. An equation (similar to 15.36) has been obtained both by LEWIS and BOWERMAN(69) and by RICHARDSON and ZAKI(83) for fluidisation with a liquid. The curve shown in Fig. 15.11 refers to the fluidisation of steel spheres in water. Even with liquid fluidisation, irregularities are obtained in the curves because of channelling and because of the formation of "bridges" of particles across the tube. It should be noted that whereas, in the absence of channelling, the pressure drop across a bed of a given expansion is directly proportional to its depth, the fluidising velocity is independent of depth.

LEVA(47), CATHALA(77) and others prefer to plot log $\frac{e^3}{(1-e)^2}$ versus log $u_c$ because $\frac{e^3}{(1-e)^2}$ is the function of voidage appearing in the KOZENY equation (11.7) for the streamline flow of a fluid through a granular bed.

### Characteristics of Gas-Solid Fluidised Systems

The ease with which a fluidised bed can be formed varies with the physical properties of the solid. Thus, such materials as the silica-alumina compounds, which are used as catalysts in the cracking process for heavy petroleum fractions, are very readily fluidised and can be maintained in that condition by the air entrained when the bed of material is gently stirred. Most materials become fluidised only during the constant upward passage of gas, however. Large particles require relatively high rates of flow of air because their terminal falling velocities are high and because the pressure drop is relatively low when a fluid flows through a bed of large particles.

The uniformity of the fluidised bed is a function of the size and the surface properties of the solids. Thus some materials tend to agglomerate, especially when the particles are less than about 50 microns in diameter, and channelling takes place. Occasionally agglomeration is so marked that fluidisation does not occur at all and when the pressure drop exceeds the weight of the particles, the whole bed is raised like a solid piston; this structure subsequently collapses and large aggregates of particles are produced.

The effect of stirring on the properties of the fluidised system has been studied by a number of workers. If the bed is stirred, an increase in volume takes place and fluidisation can be effected by the use of a smaller rate of flow of gas. Various attempts have been made to measure a property analogous to the viscosity of the bed. Thus MATHESON, HERBST and HOLT(47) measured the torque required to rotate a paddle at 200 r.p.m. in the fluidised bed. They found that it was almost directly proportional to the linear size of the particles and that it increased with increase in the density of the material. Increased aeration considerably reduced the torque required. DICKMAN and FORSYTHE(82) found that the most uniform fluidisation was obtained with those materials which showed only a small increase in apparent viscosity as the flow rate was reduced.

The tendency for the gas bubbles in a fluidised bed to coalesce and form alternate slugs of gas and fluidised solid is known to increase as the ratio of the depth of the bed to its diameter is increased. Further, the tendency for slugging is known to be dependent on the properties of the particles. Matheson *et al.* determined the maximum value of the ratio $\frac{L}{D}$ for which slugging did not take place for a number of systems. They then plotted this ratio against the apparent viscosity of the bed and obtained a curve which approximated to a straight line, as shown in Fig. 15.12. It is seen that the tendency for slugging is least with those materials which form beds whose resistance to shear is small, and thus slugging increases with the particle size of the material and its density, i.e. with terminal falling velocity.

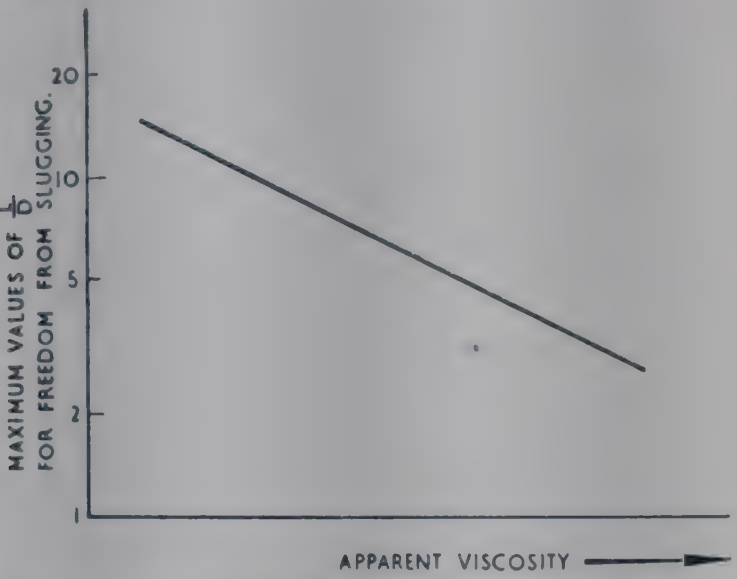


Fig. 15.12. Slugging tendency and apparent viscosity of fluidised bed

Kramers(59) has investigated the effect of rate of shear and uniformity of the bed on the apparent viscosity. He points out that a uniformly dispersed bed represents an unstable system because there is always a tendency for gas to flow from regions of relatively high solid concentration to regions of low concentration, because of the local variations in fluid pressure. Thus there is always a tendency for the bed to become non-uniform and for slugging to take place. Under equilibrium conditions on this basis, slugs are continuously formed and then break up again and disperse the solid particles. The bed is more uniform near the bottom and slug formation increases towards the top. Kramers measured the resistance to shear of fluidised beds at different rates of shear and at various distances from the bottom of the bed, using a rotating stirrer consisting of two spheres connected by a horizontal rod and rotated about a vertical axis. The resistance to shear was found to decrease with increase in the rate of shear. This effect may be partly explained by the tendency for the stirrer to entrain extra gas in the bed and thereby to increase its effective porosity. In addition, Kramers found that the resistance to shear was greater in the upper part of the bed where there was a higher degree of turbulence and where there was a tendency for slug formation to take place. The greater the degree of slugging, the higher was the apparent viscosity.

## Beds of Particles of Mixed Sizes

The most uniform fluidisation is obtained when all the particles are approximately the same size so that there is no great difference in their terminal falling velocities. If the sizes differ appreciably, elutriation occurs and the smaller particles are continuously removed from the system. If the particles forming the bed are initially of the same size, fines will be produced in time as a result of mechanical attrition or as a result of breakage due to high thermal stresses. Further, if the particles themselves take part in a chemical reaction, their sizes may alter as a result of the elimination of part of the material (e.g. during carbonisation or combustion). Any fines which are produced should be removed using a cyclone separator. Final traces of fine material can then be eliminated with an electrostatic precipitator. (See chapter 16).

LEVA(51) measured the rate of elutriation from a bed composed of particles of two different sizes. He found that, if the height of the containing vessel above the top of the bed was small, the rate of elutriation was high, but if it was greater than a certain value the rate was not affected. This was attributable to the fact that the small particles were expelled from the bed with a velocity higher than the equilibrium value in the unobstructed tube above the bed because the linear velocity of the fluid in the bed is much higher than that in the empty tube. The experiments showed that the concentration of the fine particles in the bed varied with the time of elutriation according to a law of the form

$$C = C_0 \, . \, e^{-Mt} \qquad \ldots .(15.37)$$

where $C$ is the concentration of particles at time $t$,

$C_0$ is the initial concentration of particles, and

$M$ is a constant.

The bed containing particles of different sizes behaves in similar manner to a mixture of liquids of different volatilities. Thus the finer particles, when associated with the fluidising medium, correspond to the lower boiling liquid and are more readily elutriated; their rate of removal from the system and the degree of separation are affected by the height of the reflux column. Further, a law analogous to HENRY's Law for the solubility of gases in liquids is obeyed, with the concentration of solids of given size in the bed bearing, at equilibrium, a constant relation to the concentration of solids in the gas which is passing upwards through the bed. Thus, if clean gas is passed upwards through a bed containing fine particles, these particles are continuously removed. On the other hand, if a dust-laden gas is passed through the bed, particles will be deposited until the equilibrium condition is reached. Fluidised beds are therefore sometimes used for removing suspended dusts and mist droplets from gases. They have the advantage that the resistance to flow is in many cases less than in equipment employing a fixed filter medium. Clay particles and small glass beads are used for the removal of sulphuric acid mists.

For fluidisation with a liquid, a bed of particles of mixed sizes will become sharply stratified with the small particles on top and the large ones at the bottom, provided the size range is sufficiently small for the fluid velocity to lie between the fluidising and transport velocities for each size. The pressure drop is that which would be expected for each of the layers in series.

### Countercurrent Flow

HAPPEL(47) carried out measurements on the downward flow of solid particles in a pipe in which a gas was flowing upwards. The pipe was run full of particles and they showed little tendency to move relative to one another; the porosity of the material closely corresponded with the maximum stable porosity which could be produced in a fixed bed. The condition therefore closely approximated to that of a bed on the point of fluidisation. Good agreement was obtained between calculated and measured pressure drops, for pipes of large diameter. With small diameter pipes allowance had to be made for the solid-solid friction forces at the walls.

### The Fluidised Bed Flowmeter

An interesting application of the fluidised bed is the flow meter developed by GERALD.(63) The fluidised solids are contained in a vertical graduated tube and are supported on a gauze whose apertures are slightly smaller than the particles. The most satisfactory results are obtained if the particles are approximately spherical and if the ratio of the diameters of the largest and the smallest is not greater than about 1·4. The tube should be at least ten times the diameter of the particles. The gas whose flow-rate is to be measured is passed upwards through the bed and the expansion is measured. This type of meter is useful over narrow ranges of flow rates because it gives quite a low pressure drop; it cannot be used for velocities below the fluidising velocity and becomes unsatisfactory as soon as bubbling occurs because the upper interface then oscillates too violently.

### Heat Transfer to Fluidised Beds

Fluidised systems are extensively used because of the high rates of heat transfer and the uniform temperatures within the bed, and the high coefficients for transfer from the bed to the walls of the containing vessel. Much experimental work has been carried out but a completely satisfactory correlation of the data has not so far been evolved. Several expressions of an empirical nature, in which the variables are arranged in the form of dimensionless groups, have been suggested for heat transfer to a gas-solid system but, owing to the complexity of the problem, no fundamental expressions have been derived for the heat transfer coefficients, and agreement between the results of the various workers is generally poor.

MILLER and LOGWINUK(57) obtained coefficients of between 40 and 200 lb cal/sq ft-hr-°C for heat transfer between a fluidised bed and the walls of the container. They found that the coefficients were proportional approximately to the diameter of the particles raised to the −0·6 power and the velocity to the 0·2 power.

VAN HEERDEN *et al.*(56) have obtained an empirical expression for the heat transfer coefficient which was found to be independent of the thermal conductivity of the solid particle and to vary as the −0·5 power of the Prandtl Number of the gas.

DOW and JAKOB(54) obtained heat transfer coefficients up to 170 lb-cal/sq ft-hr-°C, and these were about 100 times the values obtained for gases without suspended solids. The fluidised bed was formed in a vertical cylindrical container surrounded by a jacket containing the heating medium. Accurate measurements of temperature were made at various points. The main body of the bed was found to be at a uniform temperature with a steep temperature gradient at the entry and near the walls.

The high heat transfer coefficient at the walls of the container cannot result from the heat exchange when the solid particles hit the wall, because this would account for only a small fraction of the heat actually transferred. It is suggested that the presence of the particles near the wall breaks up the laminar sub-layer which would otherwise exist. This is further borne out by the fact that the transfer coefficient is not affected by the thermal conductivity of the solid material. The heat transfer coefficient is less when slugging occurs, presumably because there is then no longer a layer of particles near the wall throughout the whole depth of the bed.

The expression recommended by DOW and JAKOB for the calculation of heat transfer coefficients is

$$\frac{hD}{k} = 0{\cdot}55\left(\frac{D}{L}\right)^{0\cdot65}\left(\frac{D}{d}\right)^{0\cdot17}\left\{\frac{(1-e)\,\rho_s\,C_s}{e\rho C_P}\right\}^{0\cdot25}\left(\frac{DG}{\mu A}\right)^{0\cdot80} \quad \ldots.(15.38)$$

where $h$ is the heat transfer coefficient,

$k$ is the thermal conductivity of the gas,

$d$ is the diameter of the particle,

$D$ is the diameter of the tube,

$L$ is the depth of the bed,

$e$ is the voidage of the bed,

$\rho_s$ is the density of the solid,

$\rho$ is the density of the gas,

$C_s$ is the specific heat of the solid,

$C_P$ is the specific heat of the gas at constant pressure,

$\mu$ is the viscosity of the gas,

$G$ is the mass rate of flow of gas, and

$A$ is the cross-sectional area of the bed.

Other expressions have been given by LEVA(41) and VAN HEERDEN(56).

JAKOB(65) has attempted to explain differences between his results and those of VAN HEERDEN in terms of the fact that in one case the fluidised bed was being heated and in the other it was being cooled. It is likely however that this is only a partial explanation because MOLINO and HOUGEN(67) find that the heat transfer is not affected by the sense of the process.

CARR and ALMUNDSON(58) have studied the unsteady transfer of heat to a spherical particle as it enters a fluidised system in which a known amount of heat is liberated at the surface of the particle as a result of chemical reaction. A mathematical solution of the problem has been obtained for an idealised case but, in view of the fact that equilibrium conditions are rapidly reached, experimental confirmation is difficult.

## Mass Transfer in Fixed and Fluidised Beds

Experiments on mass transfer in a fixed bed have included the drying of celite catalyst particles in air, the vaporisation of naphthalene in streams of air, carbon dioxide and hydrogen, and the leaching of solutes from celite catalyst particles using water. The most satisfactory correlation of results of various workers is obtained using the *j*-factor for mass transfer $j_d$. This factor has already been discussed in Chapter 8 and has been shown to be equal to $\frac{h_D}{u_c}.\, Sc^{0\cdot67}$ (omitting the ratio $P_{Bm}/P$ which is here almost unity), where $h_D$ is the mass transfer coefficient, $u_c$ is the velocity of flow of the fluid, in this case calculated over the whole cross section of the bed, and *Sc* is the Schmidt Number for the fluid. In the calculation of the mass transfer coefficient, the whole of the surface of the particles is assumed to be available for mass transfer. WILKE and HOUGEN(27), who dried celite particles (size range approximately $\frac{1}{8} - \frac{3}{4}$ in.) in air, found that for low values of the Reynolds Group, $dG/\mu A$, (between 50 and 250), the relation between $j_d$ and the Reynolds Group was

$$j_d = 1\cdot82 \left(\frac{dG}{\mu A}\right)^{-0\cdot51} \qquad \ldots.(15.39)$$

where $d$ is the average diameter of the particles,

$G$ is the mass rate of flow of fluid through a bed of cross-section $A$, and

$\mu$ is the viscosity of the fluid.

For values of the Reynolds Group above 350, they give

$$j_d = 0\cdot989 \left(\frac{dG}{\mu A}\right)^{-0\cdot41} \qquad \ldots.(15.40)$$

Mass transfer in a fluidised system has been studied by RESNICK and WHITE(46) who measured the rate of vaporisation of naphthalene crystals of sizes between 14 and 65 mesh in streams of air, hydrogen and carbon dioxide. The experiments were carried out in a vertical tube and the particles were supported on a sintered disc. Enlargement of the cross-section of the tube at the top eliminated loss of particles by entrainment in the gas. The temperature was standardised at 25°C and gas velocities were varied between 10 and 1200 lb/sq ft-hr. The rates of vaporisation were measured by the increase in the vapour content of the gas stream and were checked by measurements of the loss in weight of the particles.

Aggregative fluidisation of the powdered material took place and at high velocities slugging occurred. In Fig. 15.13 the data are plotted for the vaporisation of particles of naphthalene of various sizes into air. It is seen that $j_d$ decreases with increase in velocity in the fixed bed; it then rises during the range over which fluidisation occurs; finally $j_d$ reaches a maximum value and then falls during the range corresponding to slugging. It should be noted, however, that whereas the *j*-factor passes through a maximum, the mass transfer coefficient continues to rise as the velocity is increased.

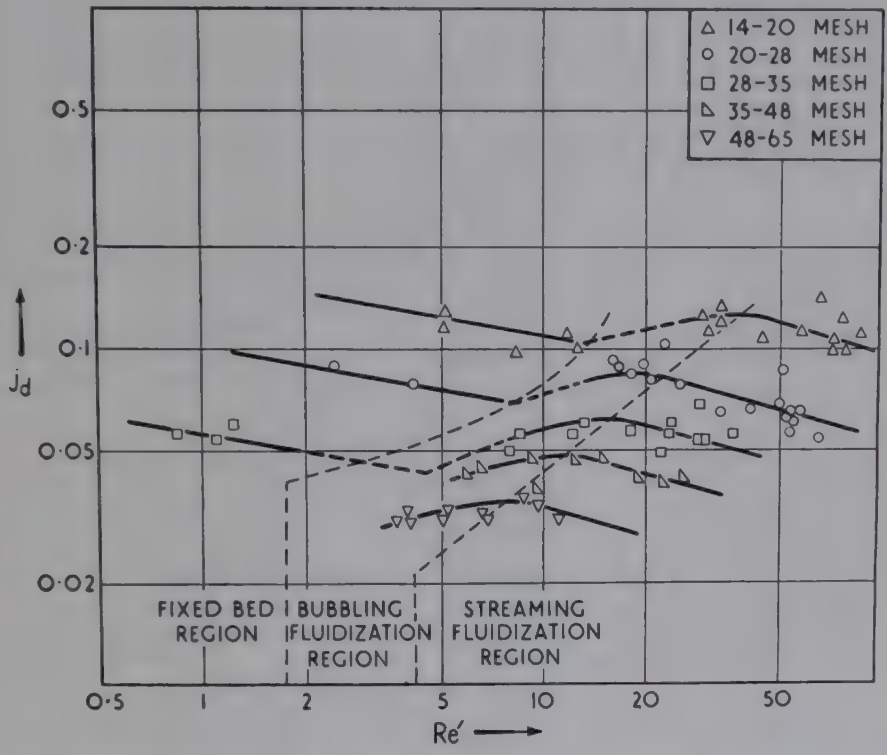


Fig. 15.13. $j_d$ for transfer of naphthalene vapour to air in fixed and fluidised beds (Resnick and White[46])

McCune and Wilhelm[47] studied the mass transfer between fluidised naphthol particles and water and also made measurements with fixed beds. Kettenring *et al*[49] evaporated water from the surface of fluidised particles of alumina gel and obtained a good correlation using the *j*-factor method. However, discrepancies were found between the results of the various workers.

Mass transfer data are conveniently expressed by Chu[78], who was able to produce a single correlation for fixed beds and fluidised beds by plotting the *j*-factor $j_d$ against a modified Reynolds Group $Re_m = \dfrac{d'' G}{A\mu(1-e)}$ (Fig. 15.14) where $d''$ is the diameter of the sphere with the same surface as the particle. By this means it was possible to cover a voidage range of from 0·25 to 0·97, with particle sizes from 0·03 to 0·5 inches. The method was equally applicable whether the fluidising medium was a liquid or a gas, and a range of Schmic Numbers from 0·6 to 1400 was thereby obtained. The recommended expressior for calculation of the mass transfer coefficient are

$$1 < Re. < 30, j_d = 5{\cdot}7\ Re_m^{-0{\cdot}78} \qquad \ldots.(15.41$$

$$30 < Re. < 5000, j_d = 1{\cdot}77\ Re_m^{-0{\cdot}44} \qquad \ldots.(15.4$$

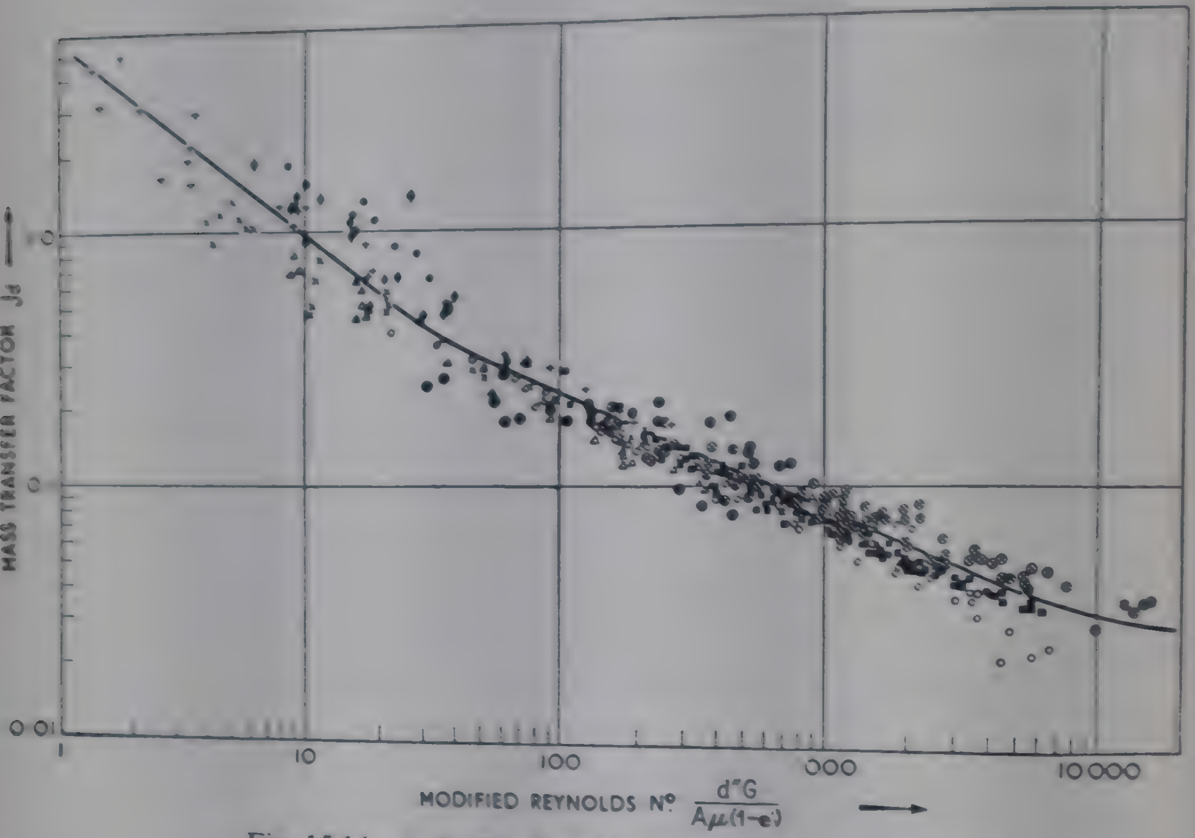


Fig. 15.14. $j_d$ for fixed and fluidised beds (CHU *et al.*[(78)])

| *Symbol* | *System* | *Schmidt No.* | *Type of particle* | *State of bed* |
|---|---|---|---|---|
| 8 | Naphthalene-air | 2.57 | Spheres, cylinders | Fixed, fluidised |
| ● | Water-air | 0.60 | Spheres, cylinders | Fixed |
| ○ | 2-naphthol-water | 1400 | Modified spheres | Fixed, fluidised |
| ● | Isobutyl alcohol-water | 866 | Spheres | Fixed |
| ◆ | Methyl ethyl ketone-water | 776 | Spheres | Fixed |
| ▲ | Salicylic acid-benzene | 368 | Modified spheres | Fixed |
| ▼ | Succinic acid—*n*-butyl alcohol | 690 | Modified spheres | Fixed |
| ⁚ | Succinic acid-acetone | 164 | Modified spheres | Fixed |

The mass transfer factor $j_d$ and the friction factor, $\dfrac{R'}{\rho u_1^{\,2}}$, divided by 3 (as giver by equation 11.11 for a fixed bed) have been plotted against the Reynold Group $Re_m$ in Fig. 15.15 and good agreement is found.

GAMSON[(52)] reports a close agreement between heat and mass transfer in fluidised system and quotes a value of 1·076 for the ratio $j_h/j_d$.

## Applications of the Fluidised Solids Technique

It has already been mentioned that the use of fluidised solids was developed very largely by the petroleum industry, where the very high heat transfer coefficients and the high degree of uniformity of temperature within the bed enabled the development of processes which would otherwise be impracticable. Fluidised solids are now used quite extensively in many other industries where it is desirable to bring about intimate contact between small solid particles and a gas stream. In many cases it is possible to produce the same degree of contact between the two phases with a very much lower pressure drop over the system. Drying of finely divided solids is now carried out in a fluidised system and some carbonisation and gasification processes, including some experimental ones for coal, are now being developed. Fluidised beds are employed in gas purification work, in the removal of suspended dusts and mists from gases, in lime burning and in the manufacture of phthalic anhydride. The use of the technique in the petroleum industry for the cracking of heavy hydrocarbons will now be described because it represents such an important application of the subject.(35)

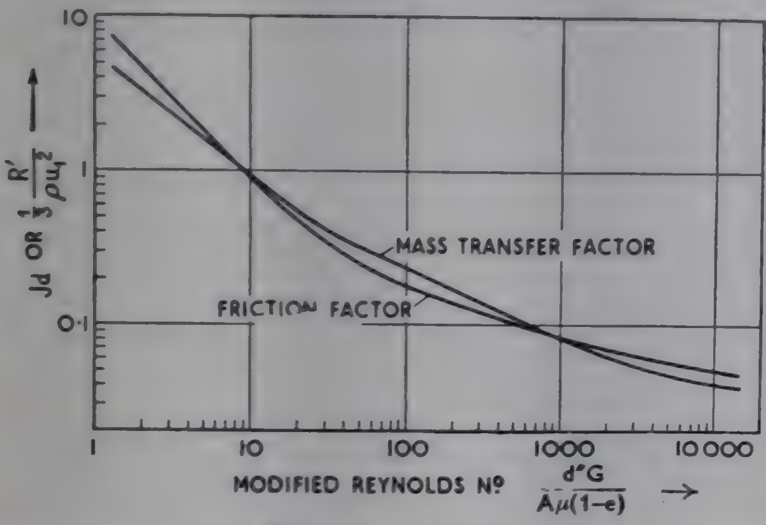


Fig. 15.15. $j_d$ and $\frac{R'}{\rho u_1^2}$ for fixed and fluidised beds (CHU *et al.*(78))

The existence of a large surplus of high boiling material after the distillation of crude oil led to the introduction of a cracking process to convert these materials into compounds of lower molecular weight and lower boiling point—in particular into petroleum spirit. The cracking was initially carried out using a fixed catalyst but local variations in temperature in the bed led to a relatively inefficient process, and the deposition of carbon on the surface of the catalyst particles necessitated taking the catalyst bed out of service periodically so that the carbon could be burned off. Many of these difficulties are obviated by the use of a fluidised catalyst since it is possible continuously to remove catalyst from the reaction vessel and to supply regenerated catalyst to the plant. The high heat transfer coefficients for fluidised systems account for very uniform temperatures within the reactors and make it possible to control conditions very closely. The fluidised system has one serious drawback; some longitudinal mixing occurs and this gives rise to a number of side reactions.

A diagram of the plant used for the catalytic cracking process is given in Fig. 15.16. Hot oil vapour, containing the required amount of regenerated catalyst, is introduced into the reactor which is at a uniform temperature of about 500°C. As the velocity of the vapour falls in the reactor, because of the greater cross-sectional area for flow, a fluidised bed is formed and the solid particles are maintained in suspension. The vapours escape from the top and the flow rate is such that the vapour remains in the reactor for about 20 seconds. It is necessary to provide a cyclone separator at the gas outlet to remove entrained catalyst particles and droplets of heavy oil. The vapour from the cracked material then passes to the fractionating unit whilst the catalyst particles and the heavy residue are returned to the bed. Some of the catalyst is continuously removed from the bottom of the reactor and, together with any fresh catalyst which is required, is conveyed in a stream of hot air into the regenerator where the carbon deposit and any adhering film of heavy oil is burned off at about 600°C: in the regenerator the particles are again suspended as a fluidised bed. The hot gases leave the regenerator through a cyclone separator, from which the solids return to the bed, and then flow to waste through an electrostatic precipitator which removes any very fine particles which are still in suspension. The temperature in the regenerator remains constant to within about 3°C even where the fluidised bed is as much as 20 feet deep and 45 feet in diameter. Catalyst is continuously returned from the regenerator to the reactor by introducing it into the supply of hot vapour. The complete time cycle for the catalyst material is about 10 minutes. By this process a product consisting of between about 50% and 75% of petroleum spirit of high octane number is obtained. The quality of the product

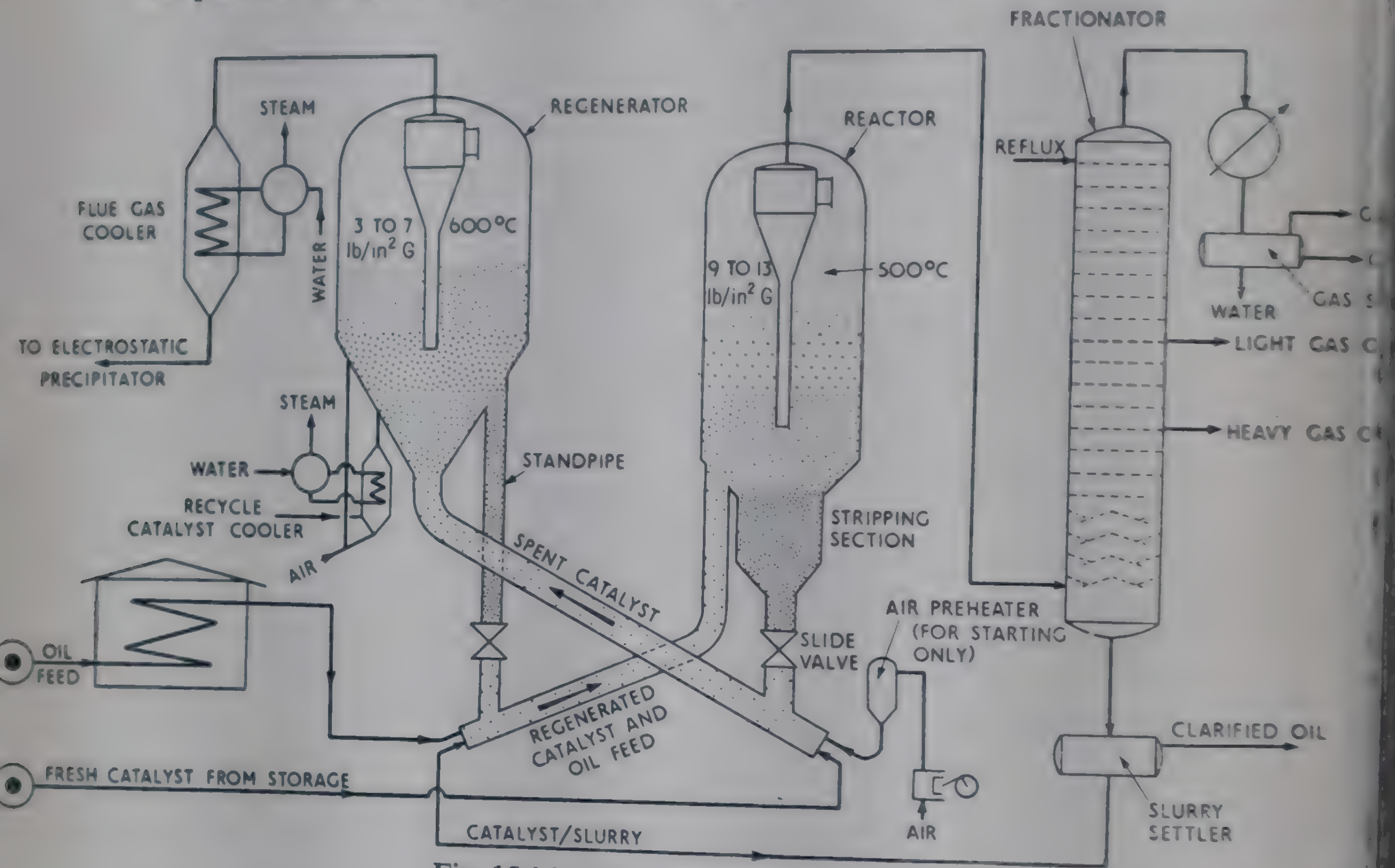

FRACTIONATOR
STEAM
REGENERATOR
REACTOR
REFLUX
FLUE GAS COOLER
3 TO 7 lb/in² G
600°C
WATER
9 TO 13 lb/in² G
500°C
C
C
WATER
GAS S
TO ELECTROSTATIC PRECIPITATOR
LIGHT GAS C
STEAM
HEAVY GAS C
WATER
STANDPIPE
RECYCLE CATALYST COOLER
STRIPPING SECTION
AIR
SPENT CATALYST
AIR PREHEATER (FOR STARTING ONLY)
SLIDE VALVE
OIL FEED
REGENERATED CATALYST AND OIL FEED
CLARIFIED OIL
FRESH CATALYST FROM STORAGE
SLURRY SETTLER
CATALYST/SLURRY
AIR

Fig. 15.16. Fluid catalytic cracking plant

can be controlled by the proportion of catalyst which is used and the exact temperature in the reactor.

From the diagram of the catalytic cracking plant given in Fig. 15.16, it will be noted that there is a complete absence of moving parts in the reactor and the regenerator. The relative positions of components are such that the catalyst is returned to the reactor under the action of gravity.

Fluidised catalysts are also used in the synthesis of high grade fuels from mixtures of carbon monoxide and hydrogen obtained either by coal carbonisation or from partial oxidation of methane.

## CONVEYING IN A FLUID

In a fluidised bed, the velocity $u_c$ of the fluid is, in general, less than the terminal falling velocity of the particles, but at high velocities the particles are transported out of the system in the fluid. Whereas fluidisation occurs only as a result of an upward flow of fluid, transport can be affected in any direction provided the fluid velocity is high enough.

Pneumatic conveying, or the transport of materials in an air stream, has been used over a long period, particularly for the conveying of grain and for the unloading of grain barges. It is now being used on an ever increasing scale and is extensively used for transporting chemicals at various works. Hydraulic conveying has long been used in the china clay industry for conveying solids from the pits to the washing and classification plants. Unlike pneumatic conveying, it can be carried out in open channels as well as in closed pipes.

Transport systems are generally operated under positive pressure with the solid particles introduced into the high pressure fluid by some mechanical feeder. In hydraulic transport it is sometimes feasible to use a slurry pump and to introduce the solids in the form of a suspension. Where the solids are to be transported over short distances, pneumatic conveying plants are frequently operated at reduced pressure as this obviates some of the difficulties experienced in introducing the solids and allows greater flexibility in operation. In reduced pressure systems the open end of the pipe dips into the solids and part of the air is entrained with the particles and part is introduced through fluidising jets.

### Mechanism of Suspension of Particles

In a vertical conveyor the method of suspension of the particles is comparatively simple since the weight of the particles is balanced by the upward force of the fluid on the particles. Impacts between the particles and the walls are infrequent compared with those in horizontal systems and therefore frictional losses due to the presence of the particles are comparatively small.

In a horizontal conveyor, the mechanism of suspension is much more complicated because the weight of the particles must ultimately be balanced by forces arising from the horizontal motion of the fluid. In most cases the supporting force will be made up of a number of components, the magnitude of each depending on the velocity and the properties of the fluid and the particles. The more important modes of suspension are as follows:—

(1) Because the fluid is normally in turbulent motion, the vertical components

of the eddies within the fluid will aid the suspension of particles, particularly if their terminal falling velocities are less than the vertical components of the eddy velocities (usually between $\frac{1}{7}$ and $\frac{1}{5}$ of the forward velocity of the fluid). Whereas a particle has an equal probability of being carried downwards or upwards by the eddy, there will always be more particles near the bottom of the tube than at the top because of the effect of gravity, and therefore the net effect of the eddy motion is to effect an upward transfer of particles.

(2) The particle will hit the walls of the pipe and, due to irregularities in the surfaces, some of the horizontal momentum may be converted into vertical momentum.

(3) Particularly if the particle is irregular in shape, it may be subjected to an upward lift due to "aerofoil effect." In Fig. 15.17, a flat plate is shown at an angle to a horizontal stream of fluid. The impact pressure of the fluid must act normally to the surface of the particle and therefore produces a horizontal and a vertical force as shown. An effect of this sort can also arise because of the velocity distribution over the cross-section of the pipe.

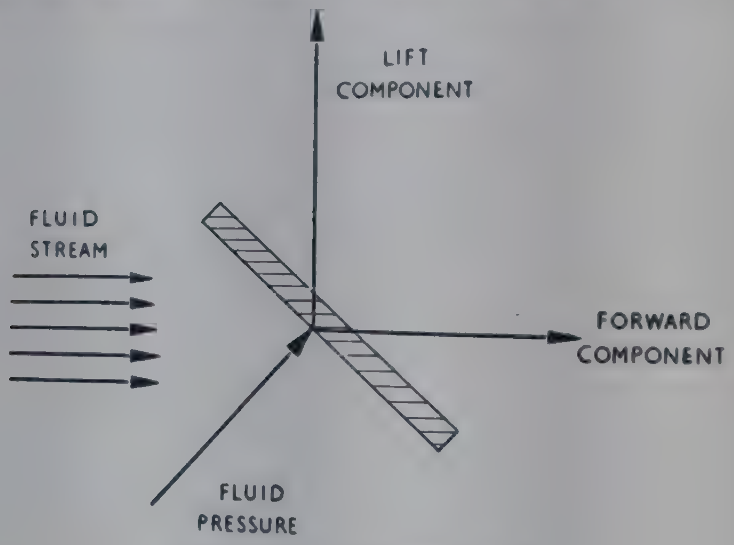


Fig. 15.17. Aerofoil effect

(4) If the particle is rotating it will be subjected to a lift on the "Magnus Principle." Fig. 15.18 shows a section through a cylinder rotating in a fluid stream. At its upper edge the cylinder and the fluid are both moving in the same direction, and at its lower edge they are rotating in opposite directions. The fluid above the cylinder is therefore accelerated and its pressure is reduced as a result of conversion of pressure energy into kinetic energy, and the pressure of the fluid below the cylinder is similarly increased. A net upward force is then produced.

## Mode of Transport

The results of visual observation on the behaviour of a 1 in. horizontal conveying system at various gas velocities is of interest. At high gas velocities, the particles appear to be fairly uniformly dispersed over the cross-section and there appears to be very little tendency for the particles to travel preferentially in the lower part of the tube. If the velocity is reduced, however, it is noticeable that the larger particles tend to segregate into the bottom portion of the tube and to move with a slightly lower velocity than the remainder. Further reduction in the velocity results first in the particles grouping themselves into clouds of relatively high solids concentration, and then subsequently the particles tend to settle out into the form of little dunes and transport of material takes place by virtue of particles moving from one dune to the next. The lower the velocity, the larger are the individual dunes, and when the dunes are higher than about half the diameter of the tube, they tend to slide along the bottom of the tube. At very low velocities, the dunes grow to such a size that they form full-bore blockages and conveying then takes place only if the material is still able to slide along the tube. In practice the plant should be operated under conditions where the particles are fairly uniformly distributed in the fluid because the risk of blockage is then a minimum.

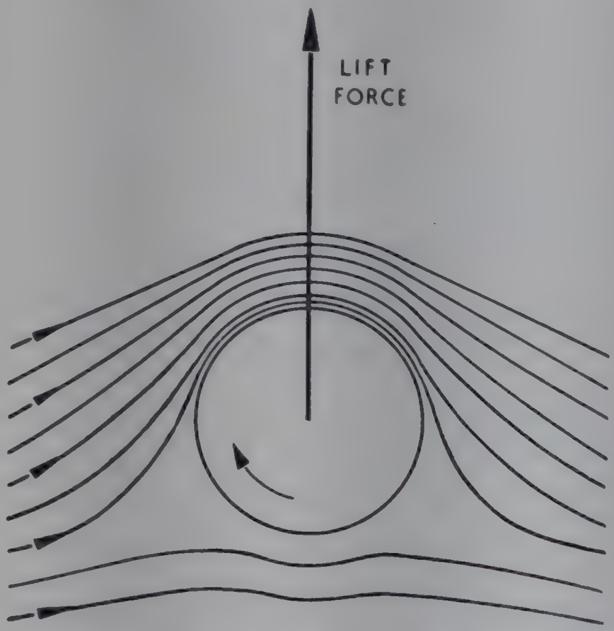


Fig. 15.18. Magnus effect

At very low rates of feed of solid material, reduction of the gas velocity may result in the formation of a bed of stationary material on the bottom of the tube; transport then takes place only in the upper portion of the tube. As the velocity is reduced, the depth of the bed is increased and it appears that the linear velocity of the gas remains approximately constant in the top of the tube. This condition can persist until only about one quarter of the cross-section of the tube is available for flow. Further reduction in the velocity results in the formation of alternate slugs of gas and solids and eventually complete blockage occurs.

In a vertical pipe, a uniform dispersion is formed at high rates of flow of gas. When the velocity is reduced, there is a tendency for alternate slugs of high and low solid concentration to be produced. Each slug moves upwards in the pipe but in so doing gives up part of its solids to the slug below. The maximum amount of downward transfer takes place near the walls where the gas velocity is lowest. The slugs remain of approximately constant size as they move upwards.

In hydraulic transport, conditions are similar to those for pneumatic conveying in a vertical pipe except that the dispersion is more even. In horizontal hydraulic conveying, a layer of solids may be deposited on the bottom of the pipe. Four regions can then be distinguished. First a fairly uniform dispersion of particles in the upper part of the tube, then a region where particles are hopping along in the fluid just above the bed, thirdly a layer of solids moving slowly along the pipe and finally, at the bottom, a stationary deposit of particles. Each of these last three regions appears in turn as the fluid velocity is reduced.

### Energy for Conveying

It is generally convenient to consider the energy requirements—and hence the pressure drop along the pipe—to be made up of two parts: first, the energy due to the motion of fluid and, secondly, the additional energy required because of the presence of the solids. Difficulties arise, however, because the fluid friction may itself be considerably modified by the presence of the solids for the following reasons:

(a) The cross-section available for the flow of the fluid is reduced because the particles, in general, will be travelling with a lower velocity than the fluid. The fraction of the total area of the duct available for fluid flow will be equal to the proportion of the total volume of the duct which is occupied by fluid at any instant. This factor is important only where the proportion of solids is high.

(b) The degree of turbulence of the fluid may be affected by the presence of the solids. Although it has occasionally been noted that the presence of very small concentrations of solids in a liquid may actually cause a reduction in the total pressure drop, the friction is usually increased.

(c) The total pressure drop in the system, and hence the absolute pressure at any point, will normally be increased by the presence of the solids. In pneumatic conveying the fluid velocity will therefore be reduced.

The energy required to maintain the particles in motion can be considered under three headings:

(1) If the particles are introduced from a hopper they will have a lower forward velocity than the fluid and therefore have to be accelerated. Because the relative velocity is greatest near the feedpoint and progressively falls as the particles are accelerated, their velocity will initially increase rapidly and, as the particles approach their limiting velocities, the acceleration will become very small. The pressure drop due to acceleration is therefore greatest near the feed point. In pneumatic conveying, the air is expanding continuously along the line and therefore the solid velocity is also increasing.

(2) Work must be done on the particles against the action of the earth's gravitational field. This is readily calculated for vertical conveying, but in horizontal conveying conditions are more complex because the particles must be intermittently lifted off the bottom of the tube.

(3) Frictional losses arise from impacts between the particles and the walls of the pipe and from collisions between particles. The former are responsible for the greater part of the losses because the relative velocity is there greatest. Collisions between particles result in appreciable losses only when the range of velocities is large, because collisions are then more frequent and more energy is dissipated at each collision.

***Energy Transfer between Fluid and Solid.*** The total rate of transfer of energy from the fluid to the particle must be equal to the net rate of increase of energy of the particle (kinetic + potential). As far as each individual particle is concerned the loss of energy is a discontinuous process, occurring only when it collides with the walls of the tube or with another particle. After each such loss of energy it will be accelerated again by the action of the fluid. Thus, even under conditions where the mean velocity of the solid particles has reached an equilibrium value, the transfer of energy to a given particle will not take place at a constant rate.

In order to provide a simplified concept of the process, it will be assumed that the fluid is moving with a constant velocity $u_f$, and each solid particle has a constant velocity $u_S$ and is freely supported in the fluid. There will then be a constant drag force $F$ acting on the particle. If this is assumed to result from form drag, from equation 14.5,

$$F = K' \,.\, \rho \,.\, (u_f - u_S)^2 \qquad \ldots.(15.43)$$

where $K'$ is a function of the shape and size of the particle. In general $K'$ will not be known, but it can be expressed in terms of the terminal falling velocity of the particle in the fluid, when in a similar state of turbulence to that in the conveying system. Thus, for a particle of mass $m$ and density $\rho_s$ settling in a fluid of density $\rho_f$ we have

$$\frac{m}{\rho_s}(\rho_s - \rho_f)g = K' \rho_f u_0^2$$

where $u_0$ is the terminal falling velocity.

Thus

$$K' = m \,.\, \frac{\rho_s - \rho_f}{\rho_s} \,.\, \frac{g}{\rho_f u_0^2} \qquad \ldots.(15.44)$$

The drag force acting on the particle in the conveyor is therefore equal to

$$m \,.\, \frac{\rho_s - \rho_f}{\rho_s} \,.\, \frac{g}{\rho_f u_0^2} \rho \, (u_f - u_S)^2 \qquad \ldots.(15.45)$$

The force acting on unit mass of particles is therefore

$$g \cdot \left(1 - \frac{\rho_f}{\rho_s}\right)\left(\frac{\rho}{\rho_f}\right)\left(\frac{u_f - u_S}{u_0}\right)^2 \qquad \ldots.(15.46)$$

If the particles are fed to the conveyor at a mass rate $W$, the mass of particles in a length $l$ of pipe is $\dfrac{W \,.\, l}{u_S}$, and the total force exerted by the fluid on the particles in a pipe of length $l$ is

$$\frac{W\,.\,l}{u_S}\cdot g\cdot\left(1-\frac{\rho_f}{\rho_s}\right)\left(\frac{\rho}{\rho_f}\right)\left(\frac{u_f-u_S}{u_0}\right)^2 \qquad \ldots.(15.47)$$

Under equilibrium conditions in a horizontal pipe, this accelerating force must be exactly balanced by the mean frictional force acting on the particles. If $\triangle P_s$ is the pressure drop in a length $l$ of pipe of cross-section $A$, attributable to the energy losses from the particles as a result of collisions,

$$\triangle P_s\,.\,A=\frac{W\,.\,l}{u_S}\cdot g\cdot\left(1-\frac{\rho_f}{\rho_s}\right)\left(\frac{\rho}{\rho_f}\right)\left(\frac{u_f-u_S}{u_0}\right)^2 \qquad \ldots.(15.48)$$

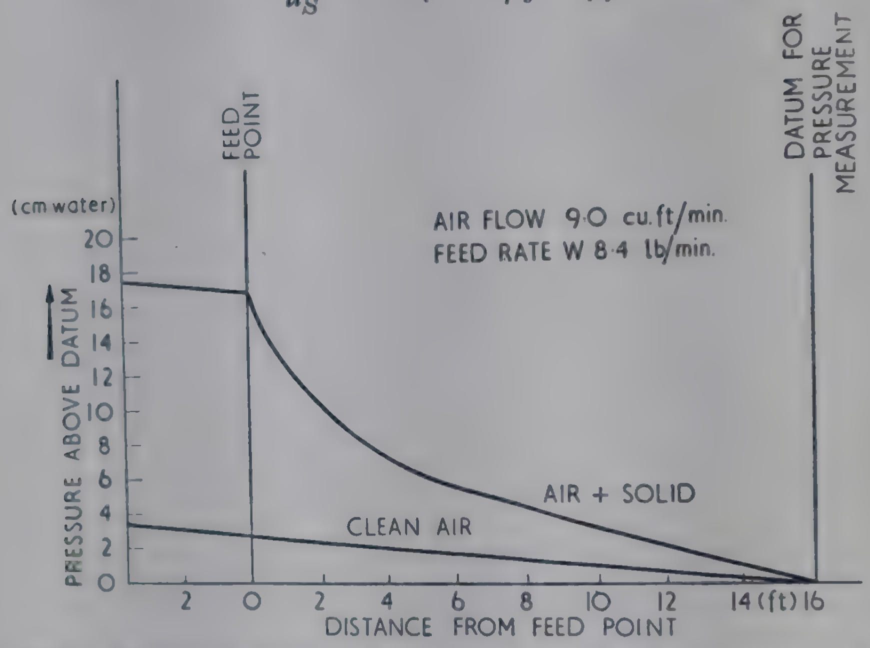


Fig. 15.19. Pressure in horizontal 1 in. conveying line for transport of cress seed

Under the corresponding conditions in a vertical pipe, the force exerted by the fluid on the particles must be equal to the sum of the weight of the particles and the frictional force. In this case, therefore,

$$\triangle P_s\,.\,A=\frac{W\,.\,l}{u_S}\cdot g\cdot\left\{\left(1-\frac{\rho_f}{\rho_s}\right)\left(\frac{\rho}{\rho_f}\right)\left(\frac{u_f-u_S}{u_0}\right)^2-\left(1-\frac{\rho}{\rho_s}\right)\right\} \qquad \ldots.(15.49)$$

It is not possible to calculate the pressure drop due to the presence of the solids directly from the above expressions unless the solid velocity $u_S$ is known. Measurements of solid velocity in a horizontal pipe have been made(68, 84) by isolating a section of the pipe using two rapidly acting shutters. The pipe between the shutters is removable and the velocity is calculated from the mass of particles in the section. Photographic methods(70) have also been used, in which two exposures are made at short intervals and the distance through which each particle has moved is then measured.

## Pressure Drops and Solid Velocities

Fig. 15.19 shows the pressure in a pneumatic conveying line plotted against distance from the feed point. and it will be noted that the pressure gradient gradually falls as the particles are accelerated and eventually attains an approximately constant value when the particles are fully accelerated. The correspondence of the regions of constant pressure gradient and of constant solid velocity has been checked experimentally.

If the total pressure drop in a horizontal pipe is plotted against the gas velocity, a curve of the form shown in Fig. 15.20 is obtained. At low velocities the pressure drop is very large because of the high concentration of solids in the tube and blockage tends to occur. As the velocity is increased, the pressure drop passes through a minimum and then increases approximately linearly with gas velocity. The minimum on the curve corresponds approximately to the velocity which would be used in practice, because lower velocities would imply a serious risk of the plant becoming blocked and high velocities would result in the use of excessive quantities of air.

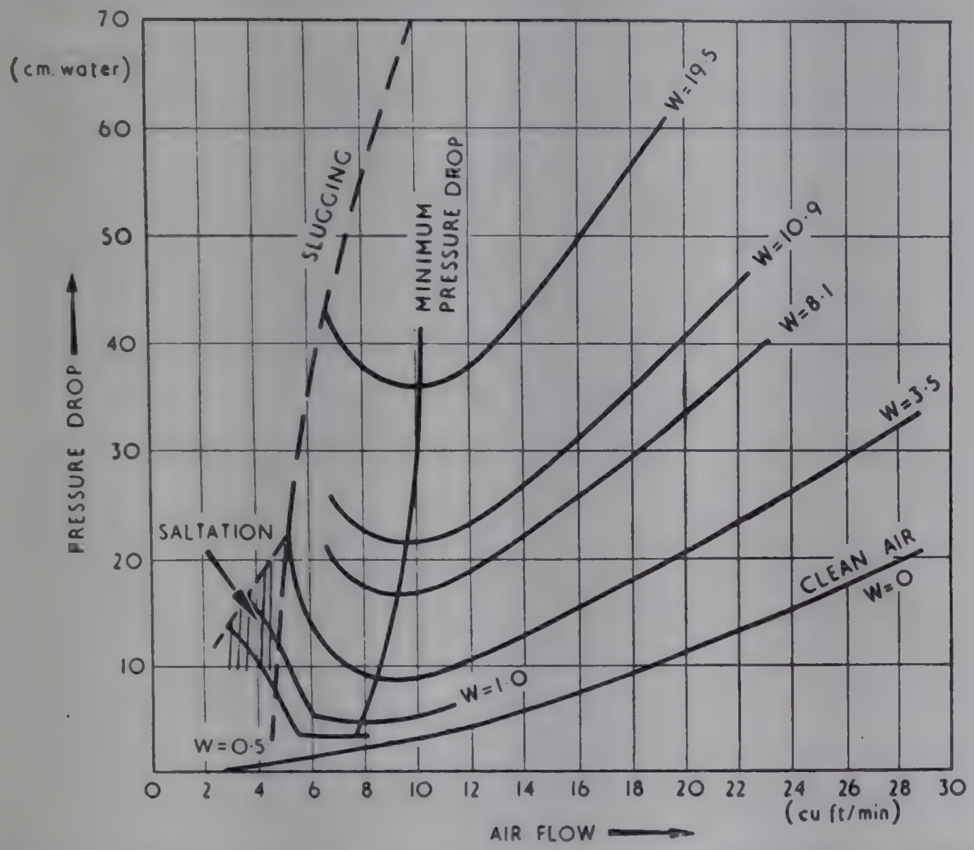


Fig. 15.20. Effect of air rate on pressure drop for transport of cress seed in 16 ft horizontal length of 1 in. pipe

The presence of bends in the conveying line will have a large effect on the energy requirements for transport of the material. The rapidity with which wear takes place on the outer wall of the tube at a bend is evidence of the large losses in energy which take place. The pressure drop over the bend is not very much greater than that obtained over the corresponding length of straight pipe. The pressure drop over the section immediately following the bend however is considerably increased because the particles have been retarded and additional energy must be supplied to accelerate them again.

Measurements(84) of the velocities of fully accelerated solids have shown that as the air velocity is increased the solid velocity generally increases so as to

6

maintain a constant relative velocity between the particles and the gas. With certain spherical particles, however, the ratio of the solid velocity to the fluid velocity is far more nearly constant.

An attempt(68) has been made to correlate the measured pressure drops with those calculated from equation 15.47. Whereas good agreement is obtained in many cases, in others the calculated pressure drop due to the solids is greater even than the overall pressure drop. This is largely attributable to the invalidity of the assumptions made in the derivation of the equation; in particular, the particles may not necessarily be freely supported in the fluid stream. More recently it has been suggested(84) that the pressure drop due to the solids is largely independent of the physical properties of the fluid and the solid particles, and is directly proportional to the weight of material in the conveying line. Experimental measurements in pneumatic conveying systems are sometimes difficult because of electrostatic effects which are obtained with a number of materials and which are capable of causing a ten-fold increase in the pressure drop due to the solids. Understanding of the problem is far from complete and considerable additional research is required.

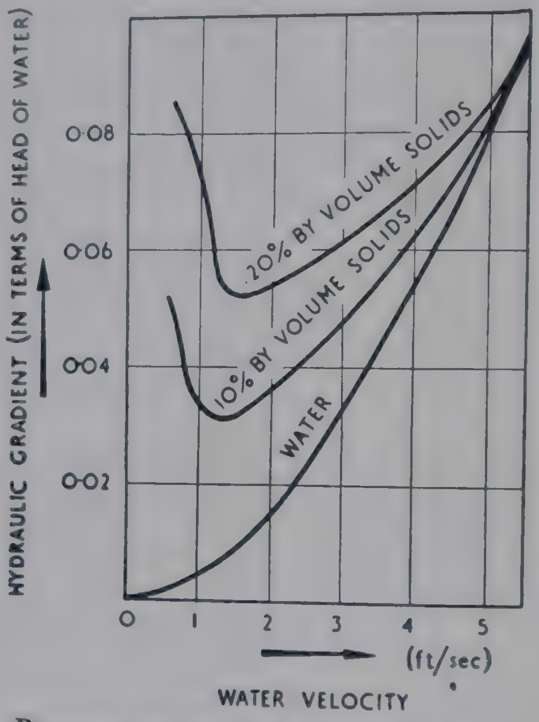


Fig. 15.21. Pressure gradient in a horizontal hydraulic conveying plant

In hydraulic transport, the difference between the solid and fluid velocities is generally very small, and only a very short distance is required for the acceleration of the particles. The pressure gradient in a horizontal hydraulic conveying plant is shown in Fig. 15.21, from which it is seen that at high velocities—for a homogeneous suspension of solid particles—the pressure is only slightly affected by the presence of the solids. As the velocity is reduced and the particles tend to be deposited on the bottom of the tube, the solids are responsible for a progressively greater proportion of the total pressure drop. At low velocities where blockage is imminent, the curves rise steeply.

## CHAPTER 16

# *Gas Cleaning*

THE necessity for removing suspended dust and mist from a gas arises not only in the treatment of effluent gas from a works before it is discharged into the atmosphere, but also in processes where solids or liquids are carried over in the vapour or gas stream. Thus in an evaporator it is frequently necessary to eliminate droplets which become entrained in the vapour, and in a plant involving a fluidised solid the removal of fine particles is necessary, first to prevent loss of material, and secondly to prevent contamination of the gaseous product. Further, in all pneumatic conveying plants, some form of separator must be provided at the downstream end.

Whereas relatively large particles with settling velocities greater than about 1 ft/sec readily disengage themselves from a gas stream, fine particles tend to follow the same path as the gas and separation is therefore difficult. In practice, dust particles may have an average diameter of about one hundredth of a millimetre and a settling velocity of about 0·01 ft/sec, so that a simple gravity settling vessel would be impracticable because of the long time required for settling and the large size of separator which would be required for a given throughput of gas.

The main problems involved in the removal of particles from a gas stream have been reviewed by ASHMAN,(61) who points out that the main reasons for removing particles from an effluent gas are:

(1) For reasons of the health of the operators in the plant and of the surrounding population. It is stated the chief danger arises from the inhaling of dust particles, and the most dangerous range of sizes is generally between about 0·5 and 3 microns.

(2) In order to eliminate explosion risks. A number of carbonaceous materials and finely powdered metals give rise to explosive mixtures with air, and flame can be propagated over large distances.

(3) In order to prevent wastage of valuable materials.

The size range of commercial aerosols and the methods available for determination of particle size and for removing the particles from the gas are shown in Fig. 16.1, which is taken from ASHMAN's paper. It will be noted that the ranges over which the various items of equipment operate overlap to some extent, and the choice of equipment will depend not only on the particle size but also on such factors as the quantity of gas to be handled, the concentration of the dust or mist, and the physical properties of the particles.

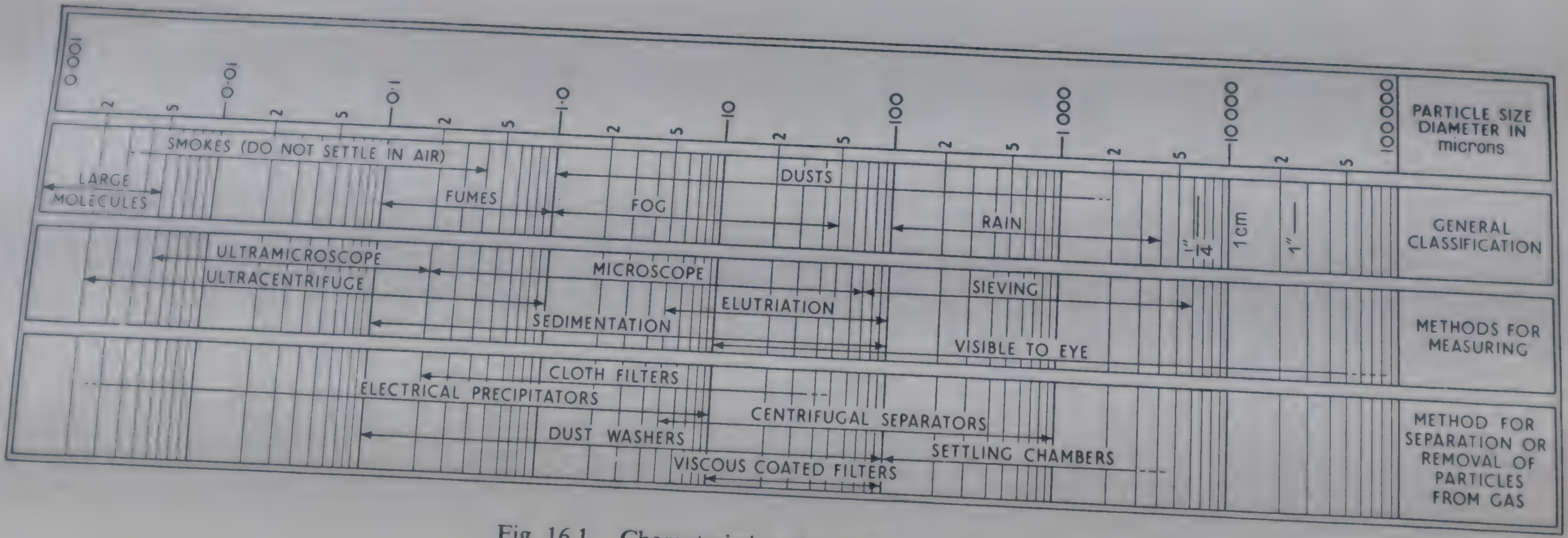


Fig. 16.1. Characteristics of aerosols and separators

Separation equipment may depend on one or more of the following principles and in some plant the relative importance of each is difficult to assess:

(1) Gravitational settling
(2) Centrifugal separation
(3) Inertia or momentum processes
(4) Filtration
(5) Electrostatic precipitation
(6) Washing with a liquid
(7) Agglomeration of solid particles and coalescence of liquid droplets.

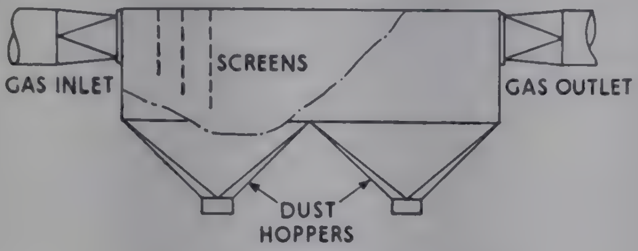


Fig. 16.2. Settling chamber

### Gravity and Momentum Separators, and Filters

If the particles are large, they will settle out of the gas stream if the cross-sectional area is increased sufficiently for the velocity to be so reduced that the eddy currents which are maintaining the particles in suspension are suppressed. In most cases, however, it is necessary to introduce baffles or screens as shown in Fig. 16.2, or to force the gas over a series of trays as shown in Fig. 16.3 which depicts a separator used for removing dust from sulphur dioxide produced by the combustion of pyrites; this equipment is suitable when the concentration of particles is high, because it is easily cleaned by opening the doors at the side. Gravity separators are seldom used now as they are very bulky and will not remove particles smaller than 50–100 microns.

Momentum separators rely on the fact that the momentum of the particles is far greater than that of the gas, so that the particles do not follow the same path as the gas if the direction of motion is suddenly changed. An apparatus devised for the separation of particles from mine gas is shown in Fig. 16.4, from which it is seen that the direction of the gas is changed suddenly at the end of each baffle. Again the separator shown in Fig. 16.5 consists of a number of vessels—up to about 30 connected in series—in each of which the gas impinges on a central baffle. The dust drops to the bottom and the velocity of the gas must be arranged so that it is sufficient for effective separation to be made without the danger of re-entraining the particles at the bottom of each vessel.

As an alternative to the use of rigid baffles, the separator may be packed with a loose fibrous material. In this case the separation will be attributable partly to gravitational settling on to the packing, partly to inertia effects and partly to filtration. If the packing is moistened with a viscous liquid, the efficiency is improved because the film of liquid acts as an effective filter and prevents the particles being picked up again in the gas stream. The Viscous filter (Fig. 16.6) consists of a series of corrugated plates, mounted in a frame and covered with a non-drying oil: these units are then arranged in banks to give the required area. They are readily cleaned and offer a low resistance to flow. Packs of slag wool offer a higher resistance but are very effective. The use of more conventional filters has already been referred to in Chapter 12. Bag filters are used

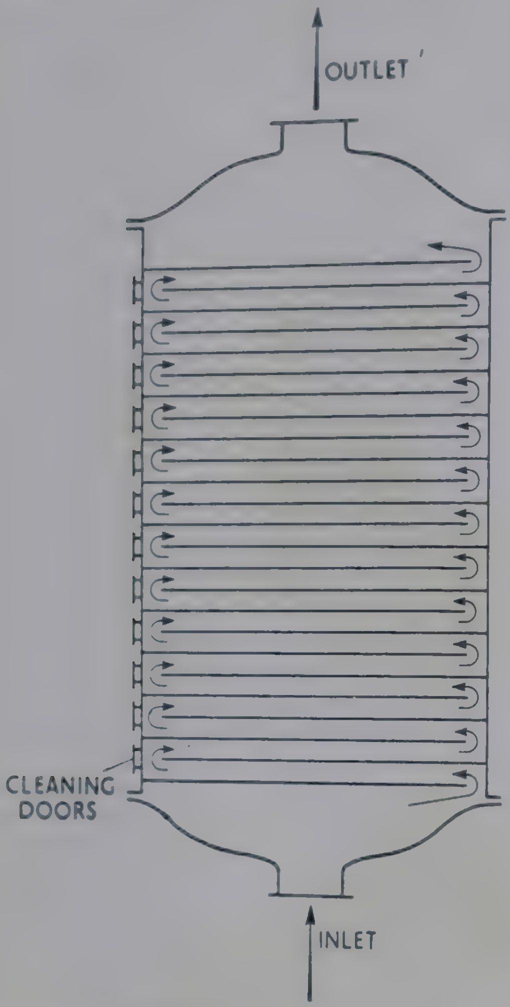


Fig. 16.3. Tray separator

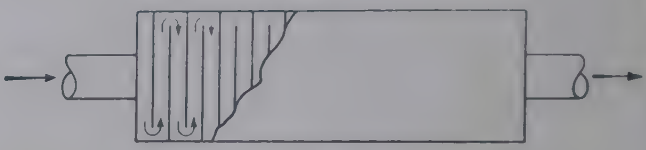


Fig. 16.4. Baffled separator

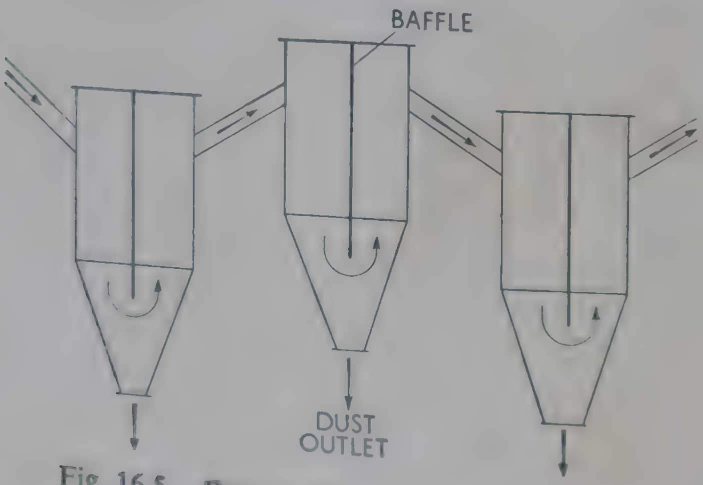


Fig. 16.5. Battery of momentum separators

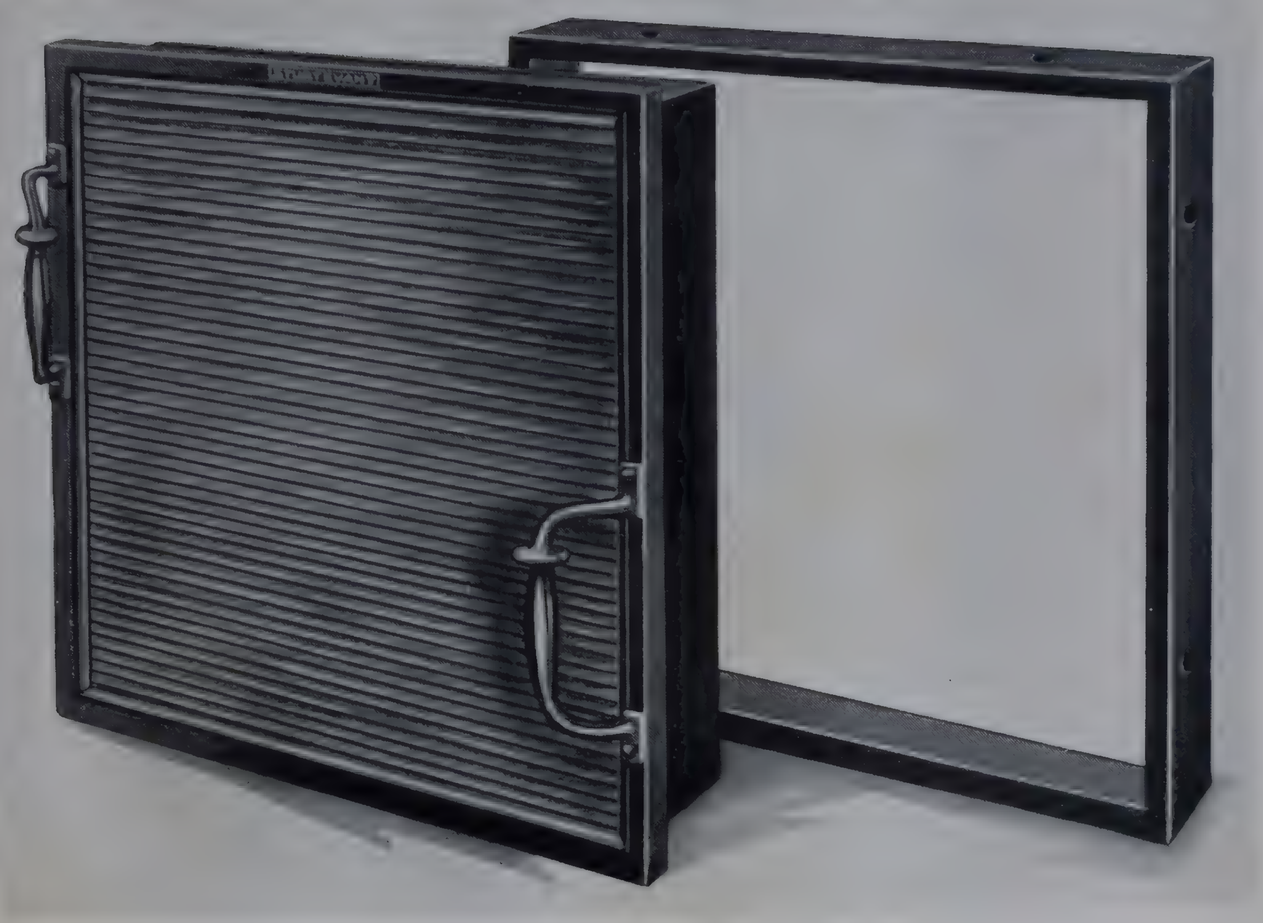


Fig. 16.6. Viscous filter

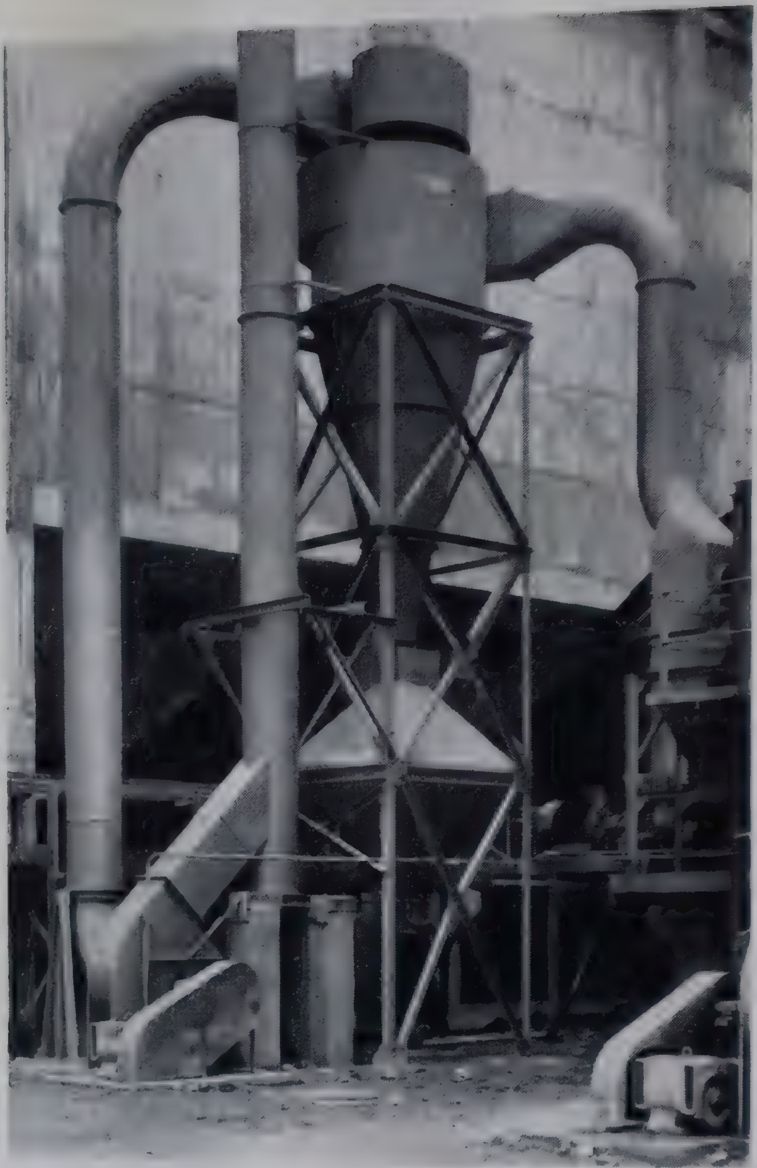


Fig. 16.9. Cyclone separator

almost exclusively for removal of dust from gas; filter presses are occasionally used in which case the cloth is covered with paper to prevent the particles from becoming embedded.

A more elaborate momentum separator is shown in Fig. 16.7. The dirty gas is fed into a conical tube with slots so arranged that the gas can escape as a result of a reversal of direction. The dust is carried forward to a cyclone separator in about 5% of the original volume of gas and the discharge is fed back again into the separator.

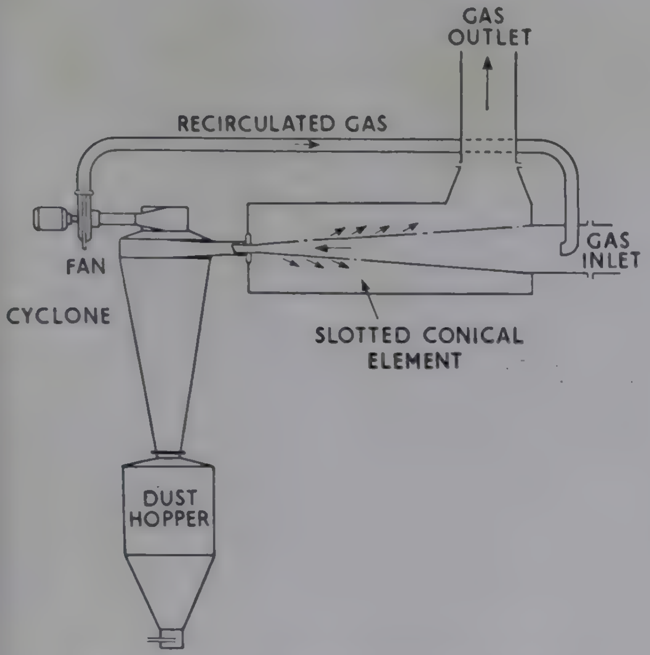


Fig. 16.7. Conical momentum separator

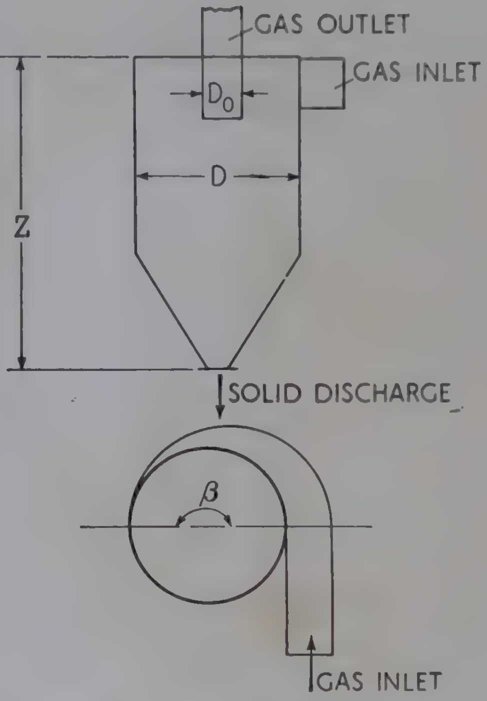


Fig. 16.8. Cyclone separator

### Cyclone Separators

The rate of settling of particles in a gas stream can be greatly increased if centrifugal rather than gravitational forces are employed. In the cyclone separator (Figs. 16.8 and 16.9) the gas is introduced tangentially into a cylindrical vessel at a velocity of about 100 ft/sec and the clean gas is taken off through a central outlet at the top. The solids are thrown outwards against the cylindrical wall of the vessel, and then move away from the gas inlet and are collected in the conical base of the plant. This separator is very effective unless the gas contains a large proportion of particles less than about 10 microns in diameter and is equally effective when used either with dust- or mist-laden gases. It is now the most commonly used general purpose separator.

The effects of the dimensions of the separator on its efficiency have been studied experimentally by TER LINDEN(43) and STAIRMAND(42), (60), who have studied the flow pattern in a glass cyclone separator with the aid of smoke injected into the gas stream. The gas is found to move spirally downwards, gradually approaching the central portion of the separator, and then rises to leave through the central outlet at the top. The magnitude and direction of the gas velocity have been explored by means of small pitot tubes, and the pressure distribution has been measured. The tangential component of the velocity of the gas appears to predominate throughout the whole depth, except within a highly turbulent central core of diameter about 0·4 times that of the gas outlet pipe. The radial component of the velocity acts inwards, and the axial component is away from the gas inlet near the walls of the separator but is in the opposite direction in the central core. Pressure measurements indicate a relatively high pressure throughout, except for a region of reduced pressure corresponding to the central core. Any particle is therefore subjected to two opposing forces in the radial direction, the centrifugal force which tends to throw it to the walls and the drag of the fluid which tends to carry the particle away through the gas outlet. Both of these forces are a function of the radius of rotation and of the size of the particles, with the result that particles of different sizes tend to rotate at different radii. As the outward force on the particles increases with the tangential velocity and the inward force increases with the radial component, the separator should be designed so as to make the tangential velocity as high as possible and the radial velocity low. This is generally effected by introducing the gas stream with a high tangential velocity, with as little shock as possible, and by making the height of the separator large.

For a spherical particle of diameter $d$, rotating at a radius $r$, the centrifugal acceleration is $\frac{u_t^2}{r}$, where $u_t$ is the tangential component of the velocity of the gas stream, on the assumption that there is no slip between the gas and the particle in a tangential direction.

The inward drag of the fluid on the particle will produce an acceleration of

$$\frac{3\pi\mu d u_r}{\frac{1}{6}\pi d^3 \rho_s} = \frac{18\mu}{d^2 \rho_s} u_r \qquad \ldots.(16.1)$$

if the relative velocity is low (from equation 14.1),

where $\mu$ is the viscosity of the gas,

$\rho_s$ is the density of the solid, and

$u_r$ is the radial component of the velocity of the gas.

The properties of the fluid and the particle can be expressed in terms of the free falling velocity $u_0$ of the particle, where

$$u_0 = \frac{d^2 g \rho_s}{18\mu} \qquad \ldots.(16.2)$$

from equation 14.16, since $\rho$ will be small compared with $\rho_s$. Substituting from equation 16.2 in equation 16.1, the inward acceleration

$$= \frac{u_r}{u_0} g \qquad \ldots.(16.3)$$

The radius at which the particle will tend to rotate is found by equating the inward and outward accelerations,

i.e.
$$\frac{u_t^2}{r} = \frac{u_r}{u_0} g$$

or
$$u_0 = \frac{u_r}{u_t^2} \cdot rg \qquad \ldots.(16.4)$$

Thus the higher the terminal falling velocity of the particle, the greater is the radius at which it will rotate and the easier it is to separate. If it is assumed that a particle will be separated provided it tends to rotate outside the central core of diameter 0·4 $D_0$, the terminal falling velocity of the smallest particle which will be retained is found by substituting $r = 0{\cdot}2D_0$ in equation 16.4.

i.e.
$$u_0 = \frac{u_r}{u_t^2} \cdot 0{\cdot}2D_0 \cdot g \qquad \ldots.(16.5)$$

In order to calculate $u_0$, it is necessary to evaluate $u_r$ and $u_t$ for the region outside the central core. The radial velocity $u_r$ is found to be approximately constant at a given radius and to be given by the volumetric rate of flow of gas divided by the cylindrical area for flow at the radius $r$. Thus, if $G$ is the mass rate of flow of gas through the separator and $\rho$ is its density, the linear velocity in a radial direction at a distance $r$ from the centre is given by

$$u_r = \frac{G}{2\pi rZ\rho} \qquad \ldots.(16.6)$$

where $Z$ is the depth of the separator.

The tangential velocity is found experimentally to be inversely proportional to the square root of the radius at all depths. Then if $u_t$ is the tangential component of the velocity at a radius $r$, and $u_{t0}$ is the corresponding value at the circumference of the separator,

$$u_t = u_{t0}\sqrt{\frac{D}{2r}} \qquad \ldots.(16.7)$$

Further, it is found that $u_{t0}$ is approximately equal to the velocity with which the gas stream enters the cyclone separator. If these values for $u_r$ and $u_t$ are now substituted into equation 16.5, the terminal falling velocity of the smallest particle which the separator will retain is given by

$$u_0 = \frac{G}{2\pi\, 0{\cdot}2D_0\rho Z} \frac{2 \times 0{\cdot}2D_0}{D} \cdot \frac{1}{u_{t0}^2} \cdot 0{\cdot}2D_0 g$$

$$= \frac{0{\cdot}2GD_0 g}{\pi\rho ZDu_{t0}^2} \qquad \ldots.(16.8)$$

If the cross-sectional area of the inlet is $A_i$, $G = A_i\rho u_{t0}$

and
$$u_0 = \frac{0{\cdot}2A_iD_0 g}{\pi ZDu_{t0}} \qquad \ldots.(16.9)$$

A small inlet and outlet therefore result in the separation of smaller particles, but as the pressure drop over the separator varies with the square of the inlet velocity and the square of the outlet velocity[42], the practical limit is set by the permissible drop in pressure. The depth and diameter of the body should be as large as possible because the former determines the radial component of the gas velocity and the latter controls the tangential component at any radius. In general, the larger the particles, the larger should be the diameter of the separator because the greater is the radius at which they rotate. The larger the diameter the greater too is the inlet velocity which can be used without causing turbulence within the separator. The factor which ultimately settles the maximum size is, of course, the cost. Because the separating power is directly related to the throughput of gas, the cyclone separator is not very flexible though its efficiency can be improved at low throughputs by restricting the area of the inlet with a damper and thereby increasing the velocity. Generally, however, it is better to use a number of cyclones in parallel and to keep the load on each approximately the same whatever the total throughput.

Because the vertical component of the velocity in the cyclone is downwards everywhere outside the central core, the particles will rotate at a constant distance from the centre and move continuously downwards until they settle on the conical base of the plant. Continuous removal of the solids is desirable so that the particles do not get entrained again in the gas stream due to the relatively low pressures in the central core. Entrainment is reduced to a minimum if the separator has a deep conical base of small angle.

Though the sizes of particles which will be retained or lost by the separator can be calculated, it is found in practice that some smaller particles are retained and some larger particles are lost. The small particles which are retained have in most cases collided with other particles and adhered to form agglomerates which behave as large particles. Relatively large particles are lost because of eddy motion within the cyclone separator and because they tend to bounce off the walls of the cylinder back into the central core of fluid. If the gas contains a fair proportion of large particles, it is desirable to remove them in a preliminary separator before the gas is fed into the cyclone.

The efficiency of the cyclone separator is greater for large than for small particles and increases with the throughput until the point is reached where excessive turbulence is created. Fig. 16.10 shows the efficiency of collection plotted against particle size for an experimental separator for which the theoretical "cut" occurs at about 10 microns. It will be noted that an appreciable quantity of fine material is collected, largely as a result of agglomeration, and some of the coarse material is lost with the result that a sharp cut is not obtained.

The effect of the arrangement and size of the gas inlet and outlet has been investigated and it has been found that the inlet angle $\beta$ (Fig. 16.8) should be of the order of 180°. Further, the depth of the inlet pipe should be small, and a square section is generally preferable to a circular one because a greater area is then obtained for a given depth. The outlet pipe should extend downwards well below the inlet in order to prevent short-circuiting.

Various modifications can be made to improve the operation of the cyclone separator in special cases. If there is a large proportion of fine material present, a bag filter may be attached to the clean gas outlet. Alternatively, the smaller particles may be removed by means of a spray of water which is injected into the separator. In some cases, the removal of the solid material is facilitated by running a stream of water down the walls and this also reduces the risk of the particles becoming re-entrained in the gas stream. The chief difficulty lies in wetting the particles with the liquid.

Because the separation of the solid particles which have been thrown out to the walls is dependent on the flow of gas parallel to the axis rather than to the effect of gravity, the cyclone can be mounted in any desired direction. In many cases horizontal cyclone separators are used and occasionally the separator is fixed at the junction of two mutually perpendicular pipes and the axis is then a quadrant of a circle as shown in Fig. 16.11. The cyclone separator is usually mounted vertically, except where there is a shortage of headroom, because removal of the solids is more readily achieved especially if there are some large particles present.

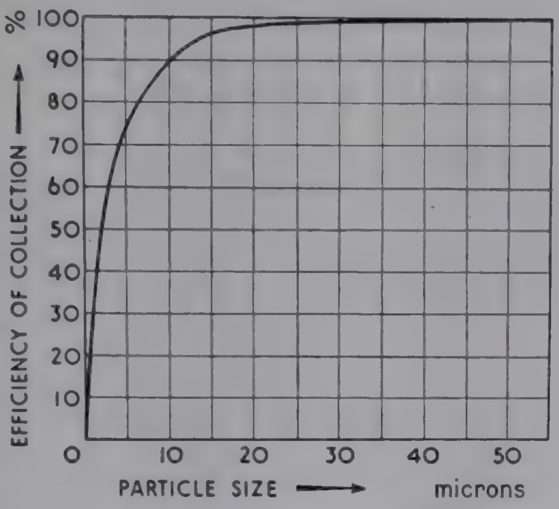


Fig. 16.10. Efficiency of a cyclone separator for various sizes of particles

A double cyclone separator is sometimes used when the range of the size of particles in the gas stream is large. It consists of two cyclone separators, one inside the other. The gas stream is introduced tangentially into the outer separator and the larger particles are deposited. The partially cleaned gas then passes into the inner separator through tangential openings and the finer particles are deposited there because the separating force in the inner separator is greater than that in the outer cylinder. In Fig. 16.12, a multicyclone is illustrated in which the gas is subjected to further action in a series of tubular units, the number of which can be varied with the throughput. This separator is therefore rather more flexible than the simple cyclone.

### Liquid Washing

If the gas contains an appreciable proportion of fine particles, liquid washing provides an effective method of cleaning which gives a gas of high purity. In

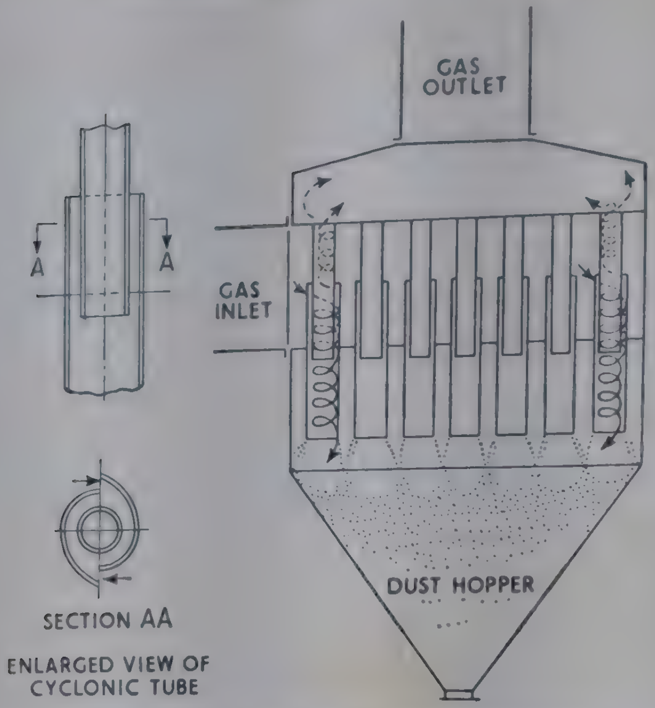

GAS OUTLET
A
A
GAS INLET
SECTION AA
ENLARGED VIEW OF CYCLONIC TUBE
DUST HOPPER

Fig. 16.12. Multi-tube cyclone separator

the spray column illustrated in Fig. 16.13, the gas flows upwards through a set of primary sprays to the main part of the column where it flows countercurrently to a water spray which is redistributed at intervals. In some cases packed columns are used for gas washing, but it is generally better to arrange the packing on a series of trays to facilitate cleaning. Fig. 16.14 shows a venturi

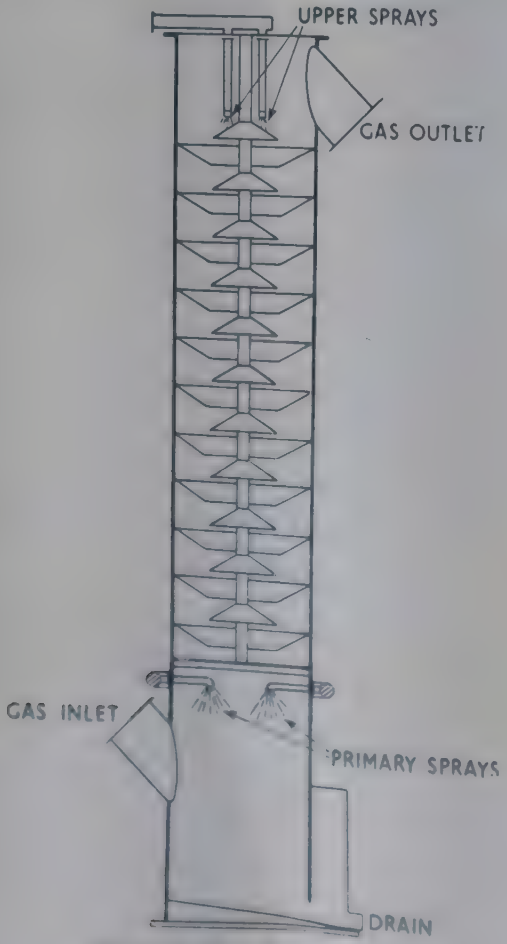

UPPER SPRAYS
GAS OUTLET
GAS INLET
PRIMARY SPRAYS
DRAIN

Fig. 16.13. Spray washer

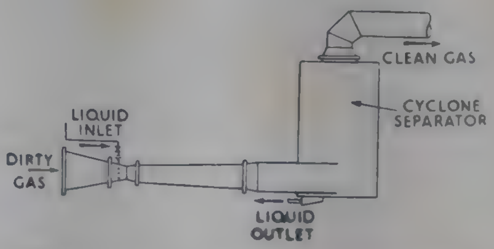

CLEAN GAS
CYCLONE SEPARATOR
LIQUID INLET
DIRTY GAS
LIQUID OUTLET

Fig. 16.14. Venturi washer with cyclone separator

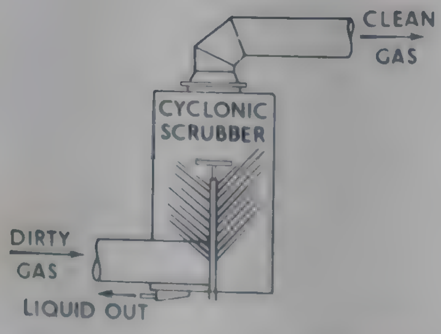

CLEAN GAS
CYCLONIC SCRUBBER
DIRTY GAS
LIQUID OUT

Fig. 16.15. Cyclonic scrubber

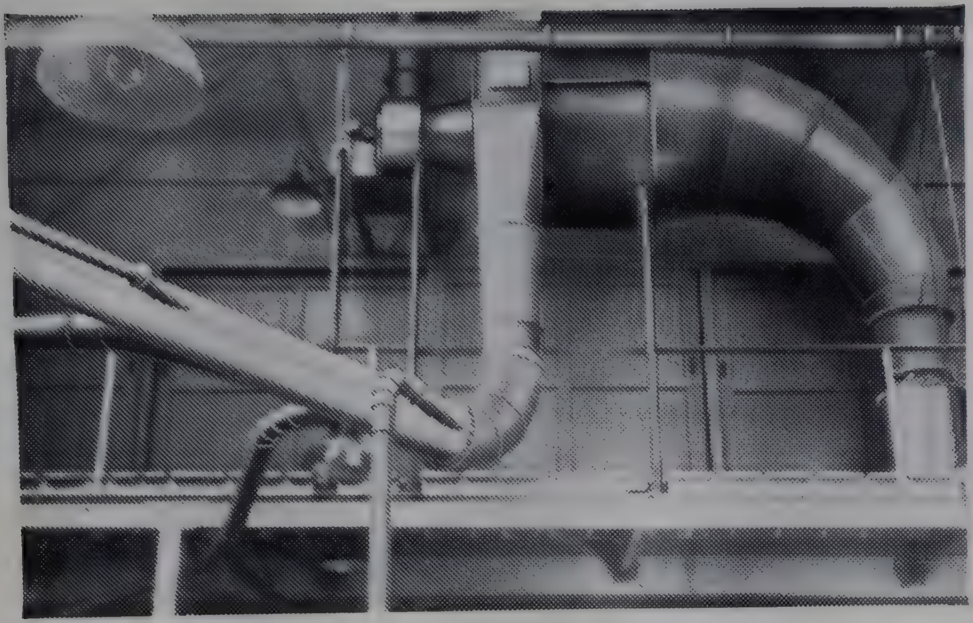


Fig. 16.11. Cyclone separator fitted in bend of duct

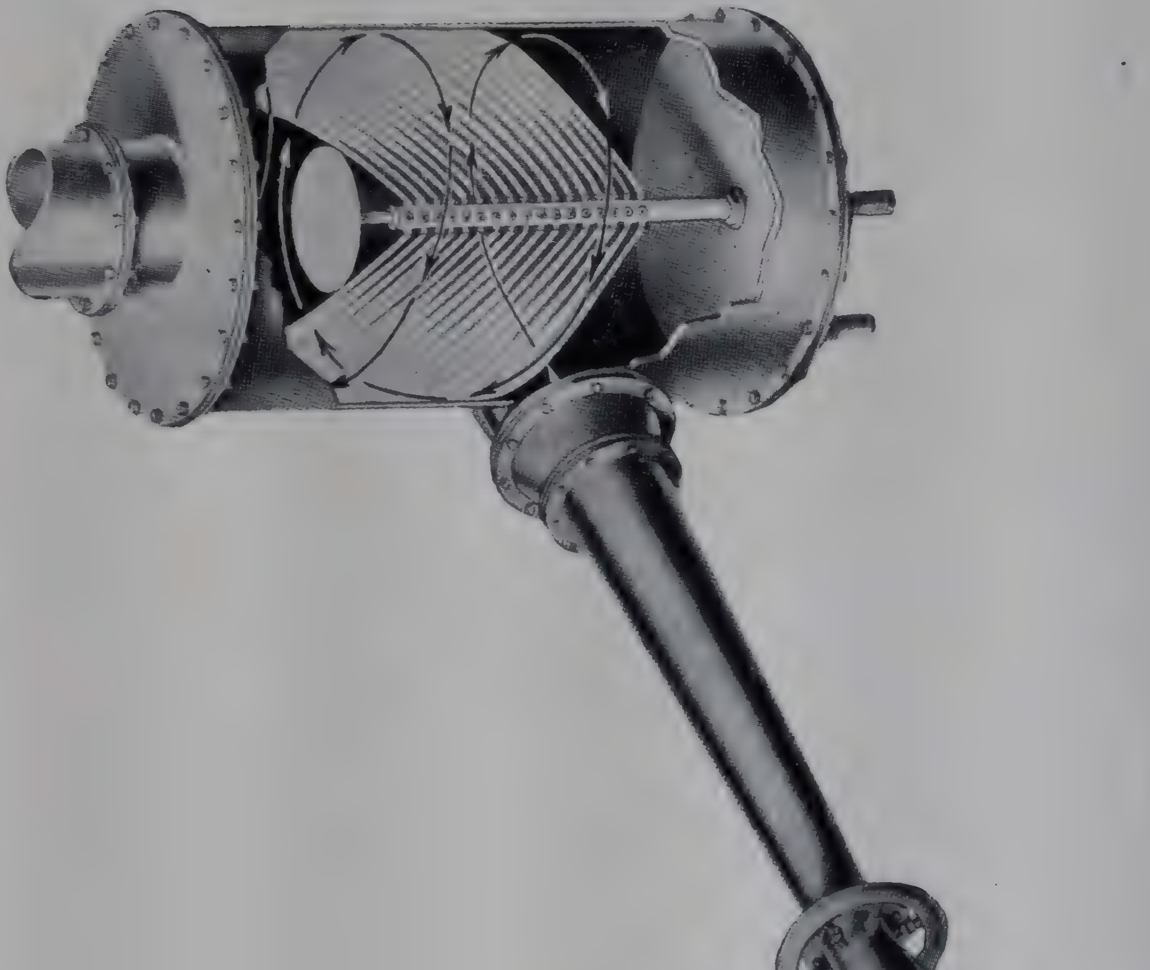


Fig. 16.16. Venturi washer with cyclonic scrubber

scrubber in which water is injected at the throat and the separation is then carried out in a cyclone separator. In the cyclonic scrubber (Fig. 16.15) the gas is introduced tangentially into a cylindrical vessel and passes upwards through a water spray. Sometimes a venturi scrubber and a cyclonic scrubber are used together as shown in Fig. 16.16. In the disintegrator gas washer (Fig. 16.17) the gas is brought into intimate contact with the scrubbing liquid by means of rotating disintegrators. The chief difficulty in operating a gas washer is wetting the particles because it is often necessary for the liquid to displace an adsorbed layer of gas. In some cases the gas is sprayed with a mixture of water and oil to facilitate wetting and it may then be bubbled through foam. This method of separation cannot be used, of course, where a dry gas is required.

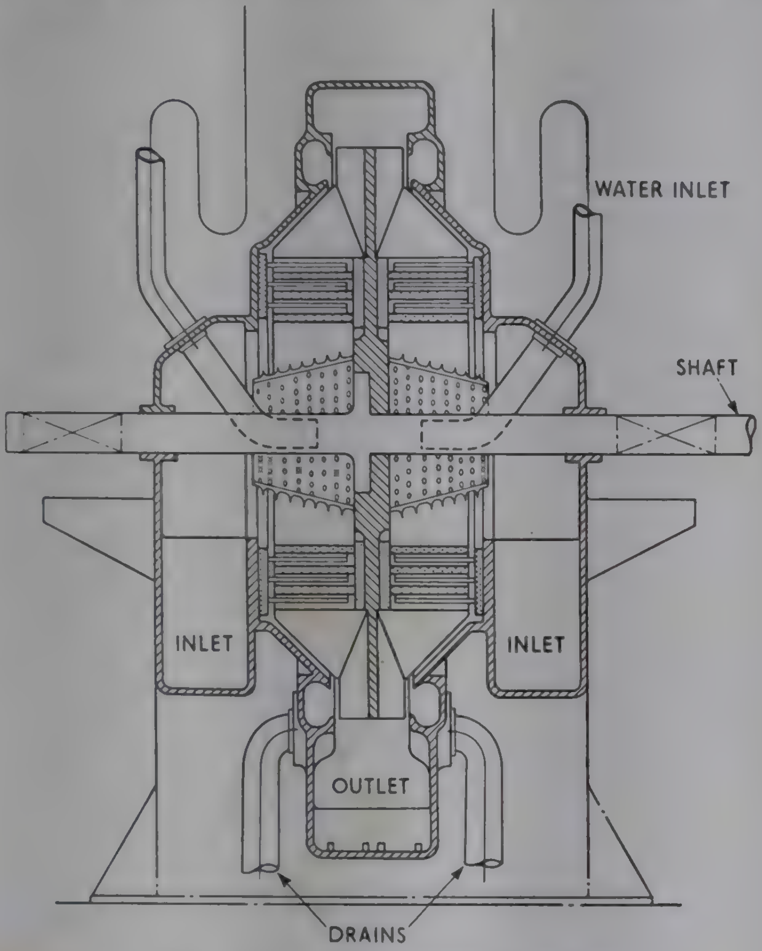


Fig. 16.17. Disintegrator washer

## Electrostatic Precipitators

When the gas contains very fine particles, an electrostatic precipitator is generally employed because its efficiency is highest when the particle size is very small. As the capital cost and running costs are relatively high it is usual to remove the coarser particles in a preliminary separator, such as a cyclone separator, and to use the electrostatic precipitator as an eliminator for the very fine material.

If the gas is passed between two electrodes charged to a potential difference of from 10,000 to 60,000 volts, it is subjected to the action of a corona discharge and ions which are given off by the smaller electrode—on which the charge

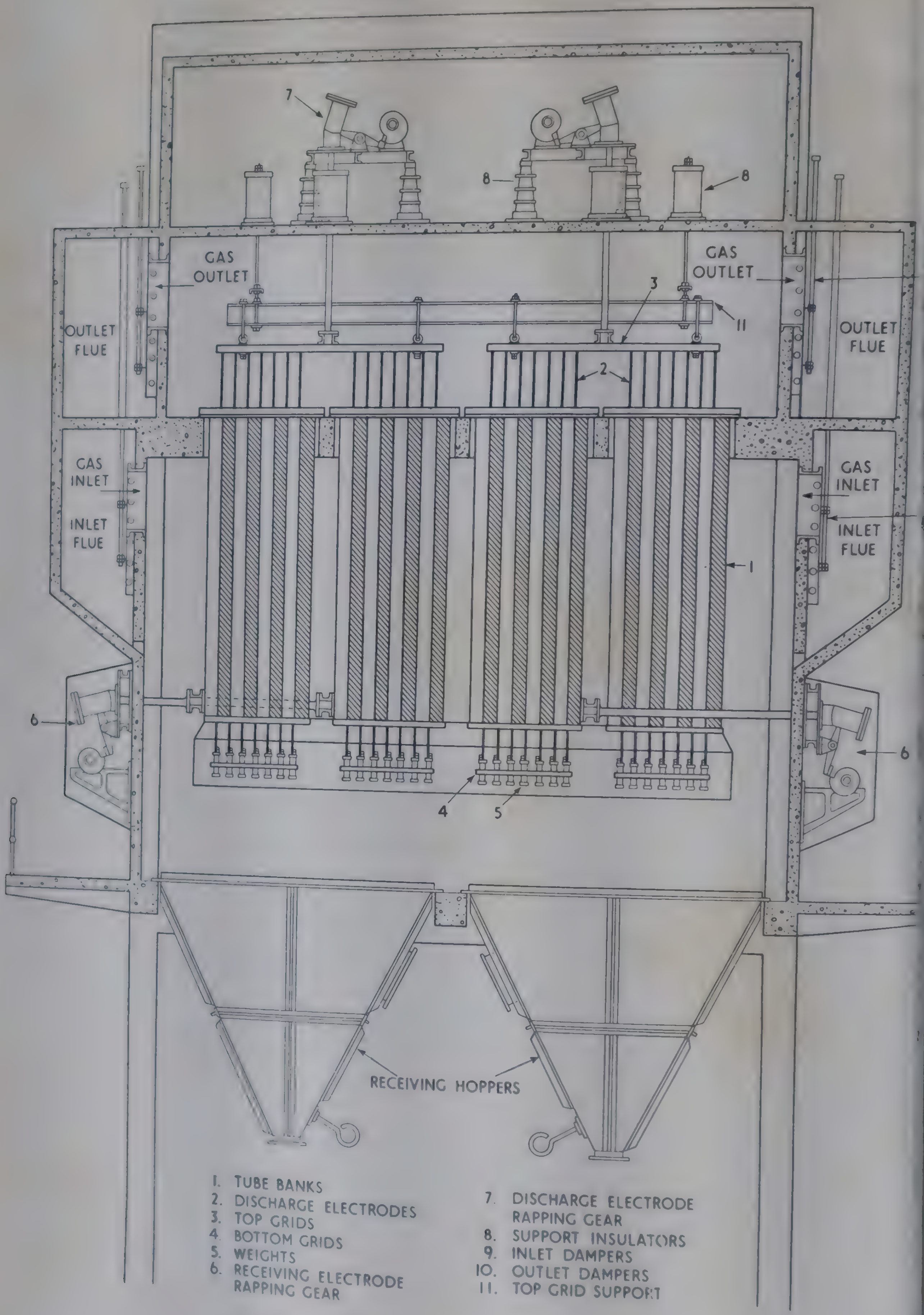
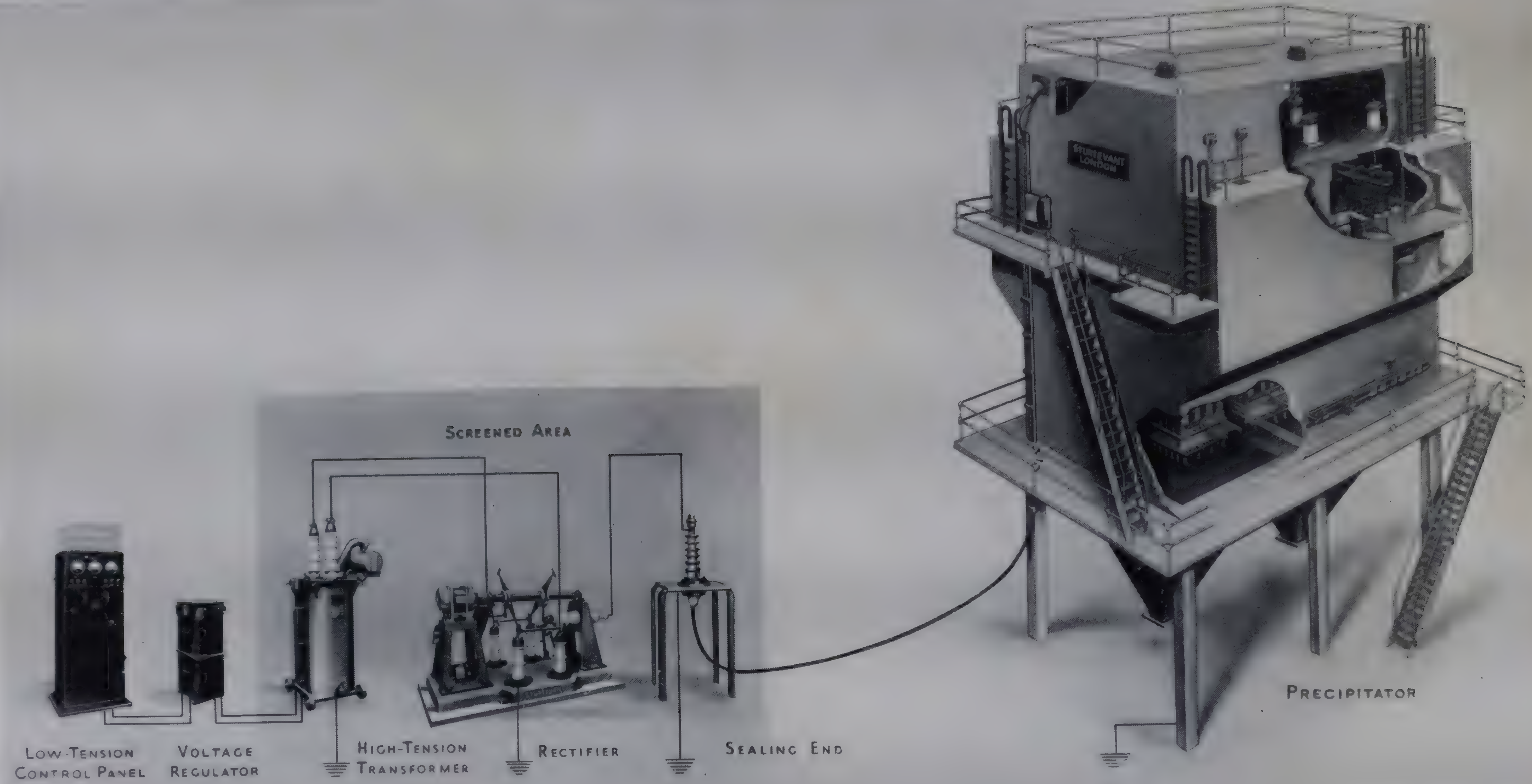
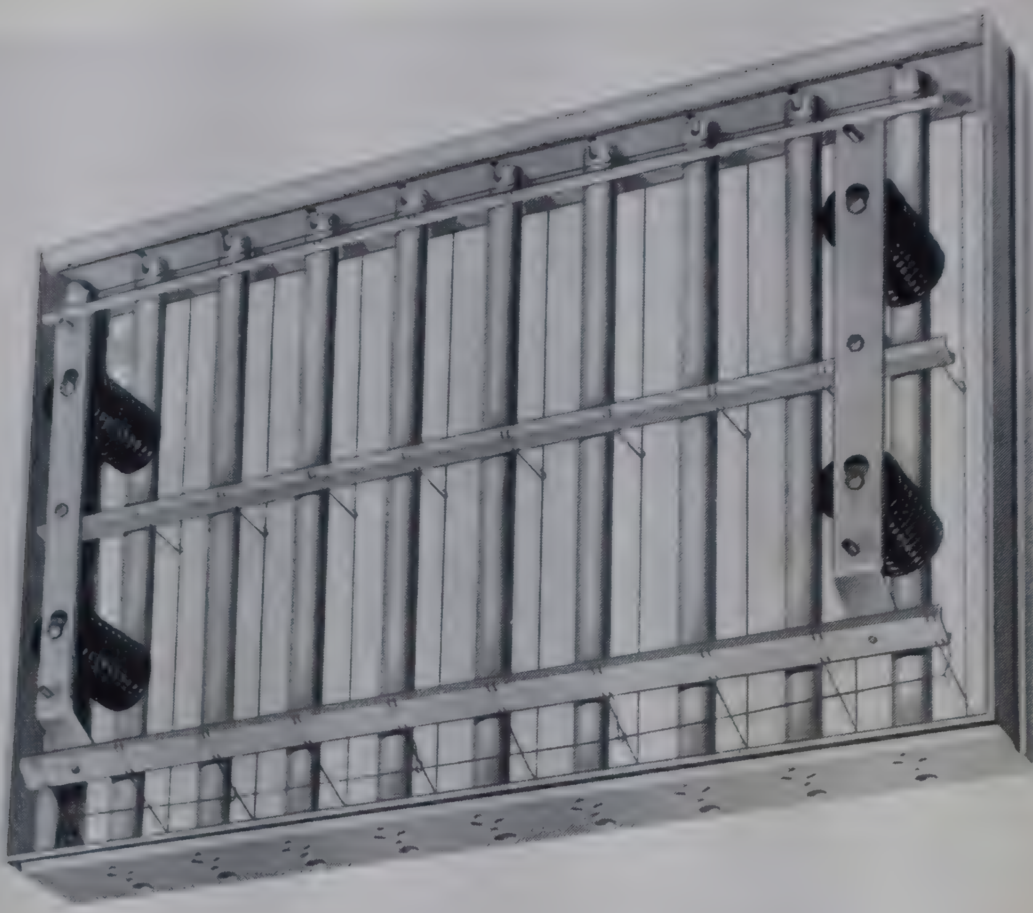

7
8
8
GAS OUTLET
GAS OUTLET
3
11
OUTLET FLUE
OUTLET FLUE
2
GAS INLET
INLET FLUE
GAS INLET
INLET FLUE
1
6
6
4
5
RECEIVING HOPPERS
1. TUBE BANKS
2. DISCHARGE ELECTRODES
3. TOP GRIDS
4. BOTTOM GRIDS
5. WEIGHTS
6. RECEIVING ELECTRODE RAPPING GEAR
7. DISCHARGE ELECTRODE RAPPING GEAR
8. SUPPORT INSULATORS
9. INLET DAMPERS
10. OUTLET DAMPERS
11. TOP GRID SUPPORT

Fig. 16.18. Pipe type electrostatic precipitator

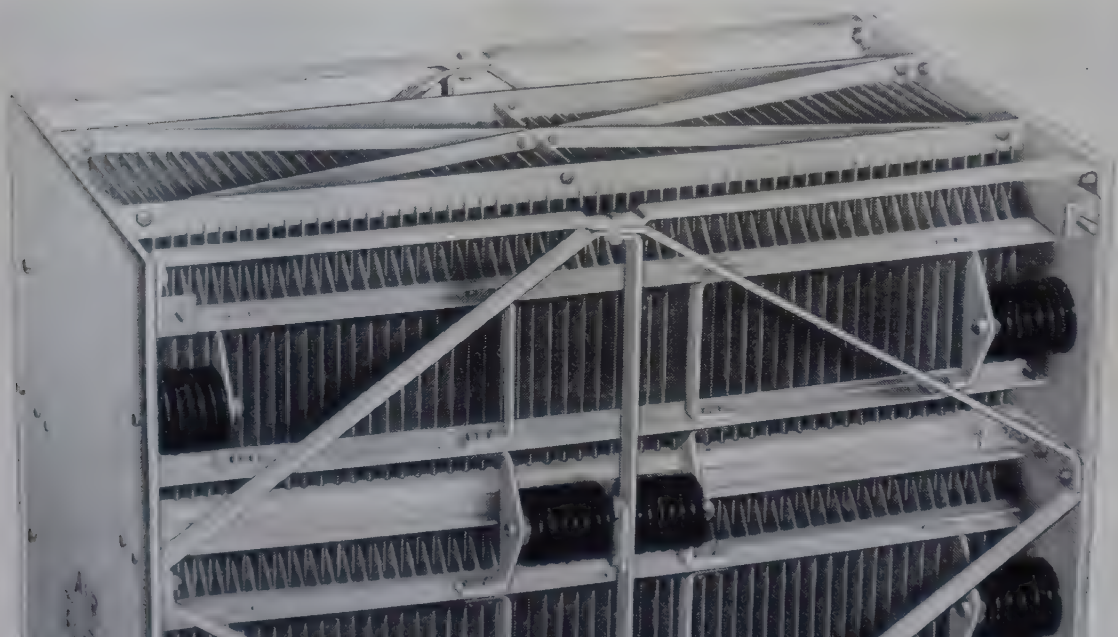

Screened Area
Sturtevant London
Low-Tension Control Panel
Voltage Regulator
High-Tension Transformer
Rectifier
Sealing End
Precipitator

Sturtevant Engineering Co. Ltd

Fig. 16.19. Perspective view of electrostatic precipitator



(*a*) Ioniser



(*b*) Collector

Fig 16.20. Plate type electrostatic precipitator

density is greater—attach themselves to the particles which are then carried to the larger electrode under the action of the electric field. The smaller electrode is known as the discharge electrode and the larger one, which is usually earthed, as the receiving electrode. Most industrial gases are sufficiently conducting to be readily ionised, the most important conducting gases being carbon dioxide, carbon monoxide, sulphur dioxide and water vapour, but if the conductivity is low, water vapour can be added. The electrodes may be in the form of plates, but the cylindrical type with the receiving electrode in the form of a metal pipe and the discharge electrode as a central wire are more efficient for large installations. The potential difference is usually determined by the tendency for arcing and, as the gap is gradually reduced as the solids or liquid droplets collect on the receiving electrode, continuous removal is desirable. This is usually achieved by rapping the electrodes, but in some cases, especially where the particles form sticky agglomerates, the electrodes are irrigated. With very sticky solids, oil vapour is sometimes injected into the gas stream so that a mobile sludge is precipitated on the electrode. A pipe precipitator is shown in Fig. 16.18 and a perspective drawing showing the high tension electrical equipment is given in Fig. 16.19.

The gas velocity over the electrodes usually varies between about 2 ft/sec and 10 ft/sec, with an average contact time of about 2 seconds. The maximum velocity is determined by the maximum distance through which any particle must move in order to reach the receiving electrode, and by the attractive force acting on the particle. This force is given by the product of the charge on the particle and the strength of the electrical field, but calculation of the path of a particle is difficult because it gradually becomes charged as it enters the field and the force therefore increases during the period of charging; this rate of charging cannot be estimated with any degree of certainty. The particle moves towards the collecting electrode under the action of the accelerating force due to the electrical field and the retarding force of fluid friction, and the maximum rate of passage of gas is that which will just allow the most unfavourably located particle to reach the collecting electrode before the gas leaves the precipitator. Collection efficiencies of nearly 100% can be obtained at low gas velocities but the economic limit is usually about 99%.

Plate type precipitators are now generally used only in small installations and the gas is often passed through an ioniser before it enters the dust collector cell. In this way the risk of arcing is reduced. Fig. 16.20*a* shows an ionising unit in which the charged electrodes at about 13,000 volts are in the form of fine wires and the earthed electrodes are metal tubes. The charged particles then pass from the ioniser to the dust collector (Fig. 16.20*b*) in which alternate plate electrodes are earthed and charged to about 6,000 volts. There is virtually no corona discharge here and the particles are simply collected on the plates.

Electrostatic precipitators are made in a very wide range of sizes and will handle up to about 100,000 cu ft of gas per minute. Although they operate more satisfactorily at low temperatures, they can be used up to about 550°C. Pressure drops over the separator are low.

### Agglomeration and Coalescence

Separation of particles or droplets is often facilitated by first effecting an increase in the effective size of the individual particles by causing them to agglomerate or coalesce, and then separating the enlarged particles. A number of methods are available. Thus, if the dust- or mist-laden gas is brought into contact with a supersaturated vapour, condensation occurs on the particles which act as nuclei. Again if the gas is brought into an ultrasonic field, the vibrational energy of the particles is increased so that they collide and agglomerate. Ultrasonic agglomeration(4, 34) has been found very satisfactory in causing fine dust particles to form agglomerates, about 10 microns in diameter, which can then be removed in a cyclone separator.

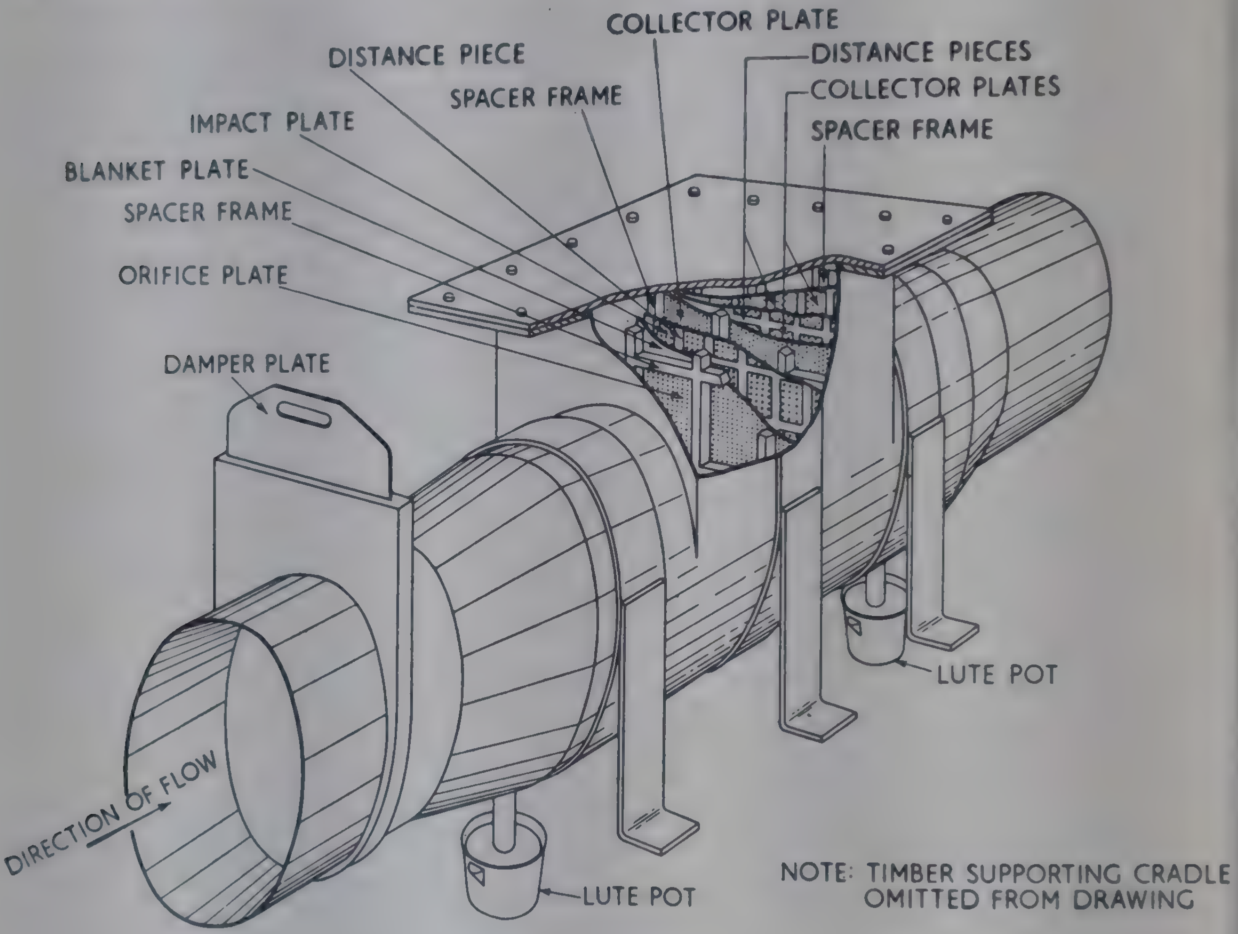

COLLECTOR PLATE
DISTANCE PIECE
DISTANCE PIECES
COLLECTOR PLATES
SPACER FRAME
SPACER FRAME
IMPACT PLATE
BLANKET PLATE
SPACER FRAME
ORIFICE PLATE
DAMPER PLATE
LUTE POT
DIRECTION OF FLOW
LUTE POT
NOTE: TIMBER SUPPORTING CRADLE OMITTED FROM DRAWING

Fig. 16.21. The Calder-Fox scrubber

***The Calder-Fox Scrubber.*** In the Calder-Fox scrubber(26), which has been developed largely for the removal of acid sprays, the gas is forced through constrictions in order to increase the turbulence and effect coalescence, and the enlarged droplets are then caused to impinge on a series of baffles.

The scrubber (Fig. 16.21) is fitted into an enlargement of the line carrying the gas, and is connected to the main portion of the duct by two conical sections.

It consists of a number of perforated plates, divided into two groups, the agglomerator plates (Fig. 16.22) and the collector plates. There are two agglomerator plates, about $\frac{1}{8}$ in. apart, which are drilled with a series of orifices at $\frac{3}{8}$–$\frac{1}{2}$ in. centres. The first is the orifice plate which has $\frac{1}{8}$ in. holes, and the other is the impact plate which has $\frac{1}{4}$ in. holes which are staggered so that there is no straight run through the two plates and the direction of flow is abruptly changed. Although the main function of these plates is to effect coalescence of the drops, some liquid does separate from the gas at this stage and is run off from the separator. Most of the separation occurs at the three collector plates which are drilled with $\frac{1}{12}$ in. holes (not staggered) and separated by $\frac{1}{8}$ in. These plates have the maximum number of holes consistent with adequate mechanical strength. The liquid which is separated here runs down over the surface of the plates as a thin film which acts as a very effective filter.

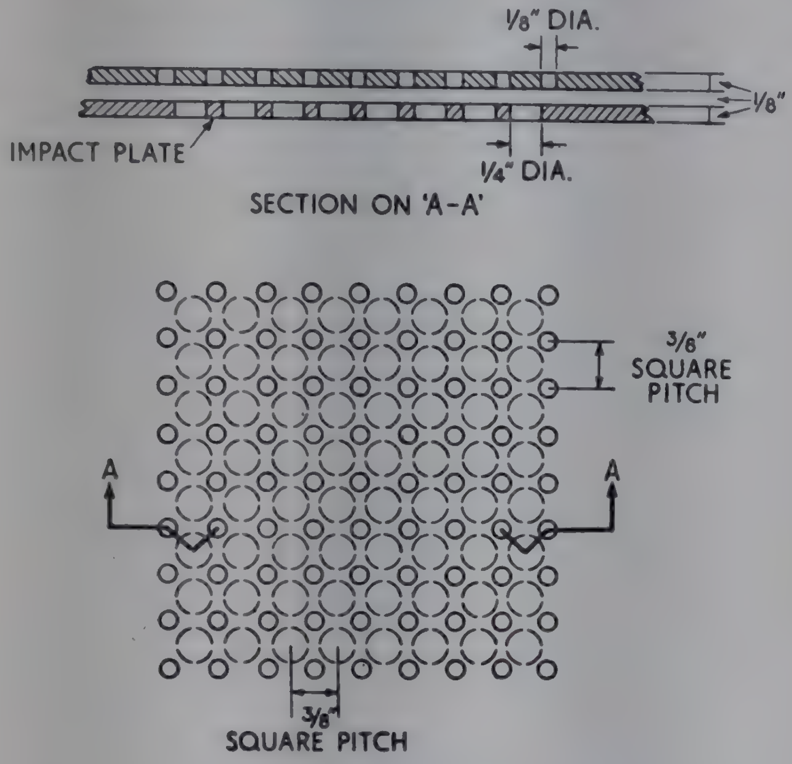


Fig. 16.22. Arrangement of impact and orifice plates

The final design of the Calder-Fox scrubber has been based on extensive preliminary investigations, during the course of which it was found that a better performance was obtained, for a given pressure drop, by using a high velocity through a short constriction rather than a lower velocity through a long constriction. In practice the velocity in the orifices is maintained at about 100 ft/sec by using a blanket plate when the throughput is reduced. The minimum size of particle which can be removed is found to be directly proportional to the distance between the orifice and impact plates, and inversely proportional to the square root of the pressure drop. A limit is set to the maximum velocity at the impact plate, not only by the permissible pressure drop, but also by the tendency for re-entrainment: this is generally greatest with liquids of low viscosities.

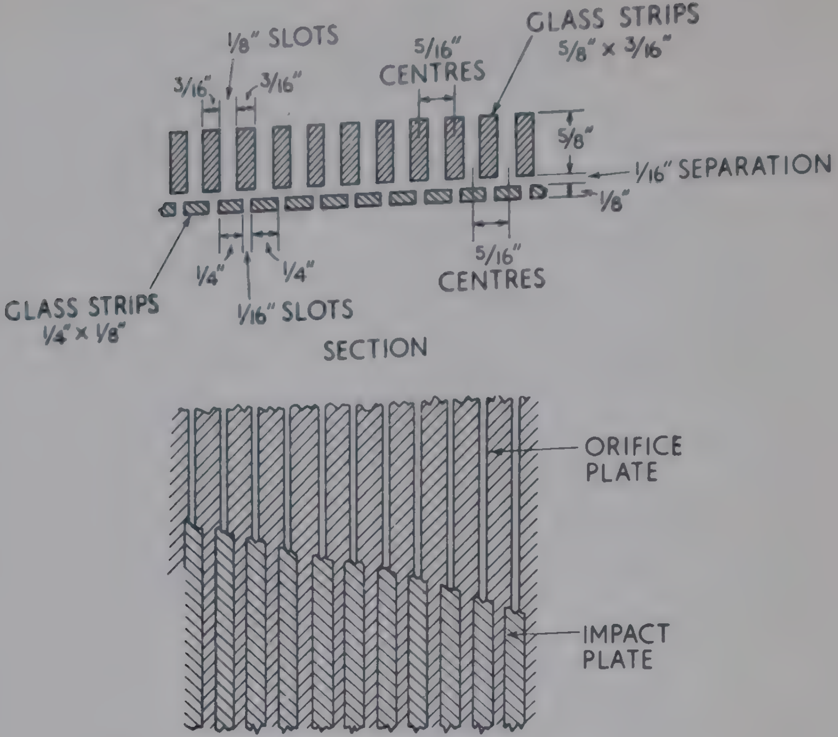


Fig. 16.23. Orifices formed from glass bars

The Calder-Fox scrubber is widely used for arresting sulphuric acid spray; scrubbers for this purpose are usually constructed of lead. When used in conjunction with hydrochloric acid plant, the orifices are formed with glass bars fixed in a corrosion resistant frame (as shown in Fig. 16.23). Droplets down to about 2 microns in diameter can be removed from the gas stream.

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## LIST OF SYMBOLS USED IN CHAPTERS 14, 15, 16

| | | |
|---|---|---|
| $A$ | Cross-sectional area of bed, pipe or thickener | $L^2$ |
| $A_i$ | Cross-sectional area of gas inlet to cyclone separator | $L^2$ |
| $A'$ | Projected area of particle in plane perpendicular to direction of motion | $L^2$ |
| $a$ | $\dfrac{18\mu}{d^2\rho_s}$ | $T^{-1}$ |
| $b$ | $\left(1 - \dfrac{\rho}{\rho_s}\right) g$ | $LT^{-2}$ |
| $C$ | Volumetric concentration of particles | — |
| $C_0$ | Volumetric concentration of particles initially | — |
| $C_p$ | Specific heat of gas at constant pressure | — |
| $C_s$ | Specific heat of solid | — |
| $c$ | $0{\cdot}33 \dfrac{\rho}{d\rho_s}$ | $L^{-1}$ |
| $D$ | Diameter of tube or vessel | L |
| $D_0$ | Diameter of outlet of cyclone separator | L |
| $d$ | Diameter of sphere or characteristic dimension of particle | L |
| $d_p$ | Mean projected diameter of particle | L |
| $d'$ | Linear dimension of particle | L |
| $d''$ | Diameter of sphere with same surface as particle | L |
| $e$ | Voidage | — |
| $F$ | Total force on particle | $MLT^{-2}$ |

| | | |
|---|---|---|
| $f$ | $\sqrt{\dfrac{b}{c}}$ | $LT^{-1}$ |
| $G$ | Mass rate of flow of fluid | $MT^{-1}$ |
| $G_E$ | Mass rate of flow required to expand bed | $MT^{-1}$ |
| $G_F$ | Mass rate of flow for fluidisation | $MT^{-1}$ |
| $g$ | Acceleration due to gravity | $LT^{-2}$ |
| $H$ | Height of slurry at time $t$ | $L$ |
| $H_c$ | Height of slurry at critical settling point | $L$ |
| $H_\infty$ | Ultimate height of slurry | $L$ |
| $h$ | Heat transfer coefficient | $ML^{-2}T^{-1}$ |
| $h_D$ | Mass transfer coefficient | $LT^{-1}$ |
| $I$ | $\displaystyle\int \frac{dt}{1 + je^{pt}}$ | $T$ |
| $i$ | Constant for given suspension | $T^{-1}$ |
| $J$ | Constant for given fluid and particle | — |
| $j$ | $\dfrac{f+v}{f-v}$ | — |
| $j_d$ | $j$-factor for mass transfer | — |
| $j_h$ | $j$-factor for heat transfer | — |
| $K$ | Constant for given shape and orientation of particle | — |
| $K'$ | Function of shape and size of particle | $L^2$ |
| $K''$ | Constant in ROBINSON's equation | — |
| $k$ | Thermal conductivity of fluid | $ML^{-1}T^{-1}$ |
| $k'$ | Constant for calculating volume of particle | — |
| $k''$ | Constant in equations for viscosity of suspension | — |
| $L$ | Depth of bed | $L$ |
| $L'$ | Distance of particle from bottom of container | $L$ |
| $l$ | Length of pipe | $L$ |
| $M$ | A constant | $T^{-1}$ |
| $m$ | Mass of particle | $M$ |
| $n$ | Index of porosity | — |
| $\Delta P$ | Pressure difference | $ML^{-1}T^{-2}$ |
| $\Delta P_s$ | Pressure drop due to solids | $ML^{-1}T^{-2}$ |
| $p$ | $2fc$ | $T^{-1}$ |
| $Q$ | Correction factor for velocity of bubble | — |
| $q$ | Constant in VAND's equation for viscosity of suspension | — |
| $R_t$ | Drag per unit area on spherical particle under terminal falling conditions | $ML^{-1}T^{-2}$ |
| $R'$ | Resistance per unit projected area of particle | $ML^{-1}T^{-2}$ |
| $r$ | Radius at which particle rotates | $L$ |
| $s$ | $(1 + je^{pt})$ | — |
| $t$ | Time | $T$ |
| $t_r$ | Retention time in thickening zone of thickener | $T$ |
| $U$ | Mass ratio of liquid to solid in feed | — |
| $u$ | Velocity of fluid relative to particle | $LT^{-1}$ |
| $u_c$ | Settling velocity of suspension or fluidisation velocity | $LT^{-1}$ |
| $u_F$ | Velocity of fluid in conveyor | $LT^{-1}$ |
| $u_i$ | Sedimentation or fluidisation velocity at infinite dilution | $LT^{-1}$ |
| $u_p$ | Velocity of particle relative to fluid | $LT^{-1}$ |
| $u_r$ | Radial component of velocity of gas in separator | $LT^{-1}$ |
| $u_S$ | Velocity of particle in conveyor | $LT^{-1}$ |
| $u_s$ | Stream velocity | $LT^{-1}$ |
| $u_t$ | Tangential component of velocity of gas in separator | $LT^{-1}$ |
| $u_{t0}$ | Tangential component of gas velocity at circumference | $LT^{-1}$ |
| $u_0$ | Free falling velocity of particle | $LT^{-1}$ |

| | | |
|---|---|---|
| $u_1$ | Velocity in pore space | $\mathrm{LT}^{-1}$ |
| $V$ | Mass ratio of liquid to solid in underflow | — |
| $v$ | Initial component of velocity of particle in *Y*-direction | $\mathrm{LT}^{-1}$ |
| $W$ | Mass rate of feed of solids to thickener or conveyor | $\mathrm{MT}^{-1}$ |
| $w$ | Initial component of velocity of particle in *X*-direction | $\mathrm{LT}^{-1}$ |
| $X$ | Average mass ratio of liquid to solid in thickening region | — |
| $x$ | Displacement of particle in *X*-direction at time *t* | L |
| $\dot{x}$ | Velocity of particle in *X*-direction at time *t* | $\mathrm{LT}^{-1}$ |
| $\ddot{x}$ | Acceleration of particle in *X*-direction at time *t* | $\mathrm{LT}^{-2}$ |
| $y$ | Displacement of particle in *Y*-direction at time *t* | L |
| $\dot{y}$ | Velocity of particle in *Y*-direction at time *t* | $\mathrm{LT}^{-1}$ |
| $\ddot{y}$ | Acceleration of particle in *Y*-direction at time *t* | $\mathrm{LT}^{-2}$ |
| $Z$ | Total height of separator | L |
| $\alpha$ | Angle between direction of motion of particle and horizontal | — |
| $\beta$ | Angle of entry into cyclone separator | — |
| $\lambda$ | Mean free path | L |
| $\mu$ | Viscosity of fluid | $\mathrm{ML}^{-1}\mathrm{T}^{-1}$ |
| $\mu_c$ | Apparent viscosity of suspension | $\mathrm{ML}^{-1}\mathrm{T}^{-1}$ |
| $\mu_l$ | Viscosity of fluid in drop or bubble | $\mathrm{ML}^{-1}\mathrm{T}^{-1}$ |
| $\rho$ | Density of fluid | $\mathrm{ML}^{-3}$ |
| $\rho_c$ | Density of suspension | $\mathrm{ML}^{-3}$ |
| $\rho_f$ | Density of fluid for terminal falling velocities | $\mathrm{ML}^{-3}$ |
| $\rho_s$ | Density of solid | $\mathrm{ML}^{-3}$ |
| *Pr.* | Prandtl number $\dfrac{C_p\mu}{k}$ | — |
| $Re._m$ | Modified Reynolds number $\dfrac{d''G}{A\mu(1-e)}$ | — |
| $Re.'$ | Reynolds number $\dfrac{ud\rho}{\mu}$ or $\dfrac{ud'\rho}{\mu}$ | — |
| *Sc.* | Schmidt number | — |

## Section G

# *Applications of Mass Transfer in Chemical Engineering*

## CHAPTER 17

# *Leaching*

THE extraction of a soluble constitutent from a solid by means of a solvent is generally referred to as leaching. The process may be employed either for the production of a concentrated solution of a valuable solid material, or in order to free an insoluble solid, such as a pigment, from a soluble material with which it is contaminated. The method used for the extraction will be determined by the proportion of soluble constituent present, its distribution throughout the solid, the nature of the solid—e.g., whether it is cellular—and the particle size.

If the solute is uniformly dispersed in the solid, the material close to the surface will first be dissolved, leaving a porous structure in the solid residue. The solvent will then have to penetrate this outer layer before it can reach further solute, and the process will become progressively more difficult and the extraction rate will become less. If the solute forms a very high proportion of the solid, this porous structure may break down almost immediately to give a fine deposit of insoluble residue, and access of solvent to the solute will not be impeded. Generally the process can be considered in three parts: first the change of phase of the solute as it dissolves in the solvent, secondly its diffusion through the solvent in the pores of the solid to the outside of the particle, and thirdly the transfer of the solute from the solution in contact with the particles to the main bulk of the solution. Any one of these three processes may be responsible for limiting the extraction rate, but the first process usually occurs so rapidly that it has a negligible effect on the overall rate.

In some cases the soluble material is distributed in small isolated pockets in a material which is impermeable to the solvent (e.g. gold dispersed in rock). In such cases the material is crushed so that all the soluble material is exposed to the solvent. If the solid has a cellular structure, the extraction rate will generally be comparatively low because the cell walls provide an additional resistance. In the extraction of sugar from beet, the cell walls perform the important function of impeding the extraction of undesirable constitutents of relatively high molecular weight and the beet should therefore be prepared in long strips so that a relatively small proportion of the cells are ruptured. In the extraction of oil from seeds, the solute is itself liquid and may diffuse towards the solvent.

The selection of the equipment for an extraction process will be influenced by the factors which are responsible for limiting the extraction rate. Thus, if the diffusion of the solute through the porous structure of the residual solids is the controlling factor, the material should be of small size so that the distance the solute has to travel is small. On the other hand if diffusion of the solute from the surface of the particles to the bulk of the solution is sufficiently slow to control the process, a high degree of agitation of the fluid is called for.

## FACTORS INFLUENCING THE RATE OF EXTRACTION

1. *The particle size.* The particle size influences the extraction rate in a number of ways. The smaller the size, the greater is the interfacial area between the solid and liquid and therefore the higher is the rate of transfer of material. Further, the smaller is the distance the solute must diffuse within the solid as already indicated. On the other hand, the surface may not be so effectively used with a very fine material if circulation of the liquid is impeded, and separation of the particles from the liquid and drainage of the solid residue are made more difficult. It is generally desirable that the range of particle size should be small so that each particle requires approximately the same time for extraction and, in particular, the production of a large amount of fine material should be avoided as it may wedge in the interstices of the larger particles and impede the flow of the solvent.

2. *The solvent.* The liquid chosen should be a good selective solvent and its viscosity should be sufficiently low for it to circulate freely. Generally a relatively pure solvent will be used initially, but as the extraction proceeds the concentration of solute will increase and the rate of extraction will progressively decrease, first because the concentration gradient will be reduced, and secondly because the solution will generally become more viscous.

3. *The temperature.* In most cases, the solubility of the material which is being extracted will increase with temperature to give a higher rate of extraction. Further the diffusion coefficient will be expected to increase with rise in temperature and this will also improve the rate. In some cases, the upper limit of temperature is determined by secondary considerations, such as the necessity of preventing enzyme action during the extraction of a solute like sugar.

4. *The agitation of the fluid.* Agitation of the solvent is important because it increases the eddy diffusion and therefore increases the transfer of material from the surface of the particles to the bulk of the solution as indicated in the following section. Further, agitation of suspensions of fine particles prevents sedimentation and more effective use is made of the interfacial surface.

## MASS TRANSFER

The mass transfer rate within the porous residue is difficult to assess because it is impossible to define the shape of the channels through which transfer must take place. However, it is possible to obtain an approximate indication of the rate of transfer from the particles to the bulk of the liquid. Using the idea of a thin film being responsible for the resistance to transfer, we can write the equation for mass transfer as

$$\frac{\mathrm{d}M}{\mathrm{d}t} = \frac{k'A(c_s - c)}{b} \qquad \ldots.(17.1)$$

where $A$ is the area of the solid-liquid interface,

$b$ is the effective thickness of the liquid film surrounding the particles,

$c$ is the concentration of the solute in the bulk of the solution at time $t$,

$c_s$ is the concentration of the saturated solution in contact with the particles,

$M$ is the mass of solute transferred in time $t$, and

$k'$ is the diffusion coefficient. (It is approximately equal to the product of the liquid phase diffusivity $D_L$ and the ratio of the total molar concentration of solvent and solute to the logarithmic mean value of the solute concentration at each side of the film (see Chapter 7). It will be taken here as approximately constant).

Considering a batch process in which $V$ is the total volume of solution which will be assumed to remain constant,

$$\mathrm{d}M = V\,\mathrm{d}c$$

and

$$\frac{\mathrm{d}c}{\mathrm{d}t} = \frac{k'A(c_s - c)}{bV}$$

The time $t$ taken for the concentration of the solution to rise from its initial value $c_0$ to $c$ is found by integration, on the assumption that both $b$ and $A$ remain constant.

Rearranging,

$$\int \frac{\mathrm{d}c}{c_s - c} = \int \frac{k'A}{Vb}\,\mathrm{d}t$$

$$\therefore \qquad \ln \frac{c_s - c_0}{c_s - c} = \frac{k'A}{Vb}\,t \qquad \text{....(17.2)}$$

If pure solvent is used initially, $c_0 = 0$, and

$$1 - \frac{c}{c_s} = e^{\frac{-k'A}{bV}t}$$

i.e.

$$c = c_s\left(1 - e^{\frac{-k'A}{bV}t}\right) \qquad \text{....(17.3)}$$

This shows that the solution approaches a saturated condition exponentially.

In most cases the interfacial area will tend to increase during the extraction, and when the soluble material forms a very high proportion of the total solid complete disintegration of the particles may occur. Although this results in an increase in the interfacial area, the rate of extraction will probably be reduced because the free flow of the solvent will be impeded and the effective value of $b$ will be increased.

Work on the rate of dissolution of regular shaped solids in liquids has been carried out by LINTON and SHERWOOD(7), and reference has already been made to this in Chapter 8. They used benzoic acid, cinnamic acid and $\beta$-naphthol as solutes, and water as solvent. For streamline flow their results were satisfactorily correlated on the assumption that transfer took place as a result of molecular diffusion alone. For turbulent flow through small tubes cast from each of the materials, the rate of mass transfer could be predicted from the pressure drop by using the "$j$-factor" for mass transfer. In their experiments with benzoic acid, they found unduly high rates of transfer because the area of the solids was increased as a result of pitting.

The effect of agitation, produced by a rotary stirrer, on mass transfer rates has been investigated by HIXSON and BAUM[(3)] who measured the rate of dissolution of pure salts in water. The degree of agitation was expressed by means of a dimensionless group $\left(\dfrac{Nd^2\rho}{\mu}\right)$ in which

$N$ is the number of revolutions per unit time of the stirrer,
$d$ is the diameter of the vessel,
$\rho$ is the density of the liquid, and
$\mu$ is its viscosity

This group will be referred to again in Chapter 26, in which the power requirements for agitators are discussed.

For values of $\dfrac{Nd^2\rho}{\mu}$ less than 67,000, the results are correlated by the equation

$$\frac{K_L d}{D_L} = 2{\cdot}7 \times 10^{-5} \left(\frac{Nd^2\rho}{\mu}\right)^{1{\cdot}4} \left(\frac{\mu}{\rho D_L}\right)^{0{\cdot}5} \qquad \ldots.(17.4)$$

and for higher values of $\dfrac{Nd^2\rho}{\mu}$,

$$\frac{K_L d}{D_L} = 0{\cdot}16 \left(\frac{Nd^2\rho}{\mu}\right)^{0{\cdot}62} \left(\frac{\mu}{\rho D_L}\right)^{0{\cdot}5} \qquad \ldots.(17.5)$$

where $K_L$ is the mass transfer coefficient, equal to $\dfrac{k'}{b}$ in equation 17.1.

Further experimental work was carried out on the rates of melting of a solid in a liquid, using a single component system, and HIXSON and BAUM were able to express their results for the heat transfer coefficient as

$$\frac{hd}{k} = 0{\cdot}207 \left(\frac{Nd^2\rho}{\mu}\right)^{0{\cdot}63} \left(\frac{C_p\mu}{k}\right)^{0{\cdot}5} \qquad \ldots.(17.6)$$

for values of $\dfrac{Nd^2\rho}{\mu}$ greater than 67,000.

Here $h$ is the heat transfer coefficient,
$k$ is the thermal conductivity of the liquid, and
$C_p$ is its specific heat.

Thus we have from equations 17.5 and 17.6 that at high degrees of agitation the ratio of the heat and mass transfer coefficients is almost independent of the speed of the agitator and

$$\frac{K_L}{h} = 0{\cdot}77 \left(\frac{D_L}{\rho C_p k}\right)^{0{\cdot}5} \qquad \ldots.(17.7)$$

PIRET[8] has attempted to reproduce the conditions in a porous solid using banks of capillary tubes, beds of glass beads and porous spheres, by measuring the rate of transfer of a salt as solute through water to the outside of the system. He has shown that the rate of mass transfer is that which would be predicted for an unsteady transfer process and has satisfactorily taken into account the shapes of the pores.

## TYPES OF EQUIPMENT

Three distinct processes are involved in the normal leaching operation. They are:

(1) Dissolving the soluble constituent.

(2) Separating the solution, so formed, from the insoluble solid residue.

(3) Washing the solid residue in order to free it of unwanted soluble matter or to obtain, as product, as much of the soluble material as possible.

Leaching has in the past been carried out mainly as a batch process but many continuous plants are now being developed. The type of equipment employed will depend on the nature of the solid—whether it is granular or cellular and whether it is coarse or fine. The normal distinction between coarse and fine solids is that the former have sufficiently large settling velocities for them to be readily separable from the liquid, whereas the latter can be maintained in suspension with the aid of only a small amount of agitation. Generally the solvent can be allowed to percolate through beds of the coarse materials, whereas the fine solids offer too high a resistance.

As already pointed out, the rate of extraction will, in general, be a function of the relative velocity between the liquid and the solid. In some plants the solid is stationary and the liquid flows through the bed of particles; in some continuous plants the solid and liquid move countercurrently.

### Extraction from Cellular Materials

With seeds such as soya beans, containing only about 15% of oil, solvent extraction is often used because mechanical methods are then not very efficient. Light petroleum fractions are generally used as solvents; trichlorethylene has been used where fire risks are serious, and acetone or ether where the material is very wet. A batch plant for the extraction of oil from seeds is illustrated in Fig. 17.1 and consists of a vertical cylindrical vessel divided into two sections by a slanting partition. The upper section is filled with the charge of seeds which is sprayed with fresh solvent from a distributor. The solvent percolates through the bed of solids and drains into the lower compartment where, together with any water extracted from the seeds, it is continuously boiled off by means of a steam coil. The vapours are taken to an external condenser and the mixed liquid is passed to a separating box from which the solvent is continuously fed back to the plant and the water is run to waste. By this means a concentrated solution of the oil is produced by the continued application of pure solvent to the seeds.

The Bollmann continuous moving bed extractor(4) (Fig. 17.2) consists of a series of perforated buckets, arranged as in a bucket elevator, contained in a vapour-tight vessel, and is widely used with seeds which do not disintegrate on extraction. Solid is fed into the top bucket on the downward side and is discharged from the top bucket on the upward side, as shown in Fig. 17.3. The solvent is sprayed on to the solid which is about to be discarded, and passes downwards through the buckets so that countercurrent flow is achieved. The solvent is then finally allowed to flow down through the remaining buckets in co-current flow. A typical extractor will move at about 1 revolution per hour, each bucket containing about 800 lb of seeds. Generally about equal weights of seeds and solvent are used and the final solution, known as miscella, contains about 25% of oil.

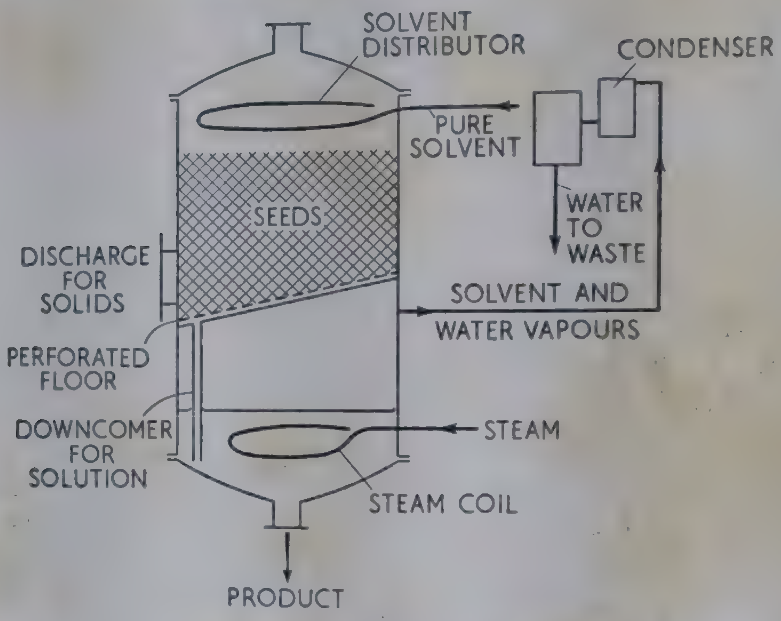

Fig. 17.1. **Batch plant for extraction of oil from seeds**

The Bonotto extractor(4) consists of a tall cylindrical vessel with a series of slowly rotating horizontal trays. The solid is fed continuously on to the top tray near its outside edge and a stationary scraper, attached to the shell of the plant, causes it to move towards the centre of the plate. It then falls through an opening on to the plate beneath, and another scraper moves the solids outwards on this plate which has a similar opening near its periphery. By this means the solid is moved across each plate, in opposite directions on alternate plates, until it reaches the bottom of the tower from which it is removed by means of a screw conveyor. The extracting liquid is introduced at the bottom and flows upwards so that continuous countercurrent flow is obtained, but a certain amount of mixing of solvent and solution takes place when the density of the solution rises as the concentration increases.

The recently developed continuous extractor illustrated in Fig. 17.4 consists of a cylindrical tank in which 18 sector-shaped perforated cells slowly rotate. The material to be extracted is continuously fed in at a fixed point at such a rate that a cell is filled in the time taken to pass the feeder. It is then sprayed

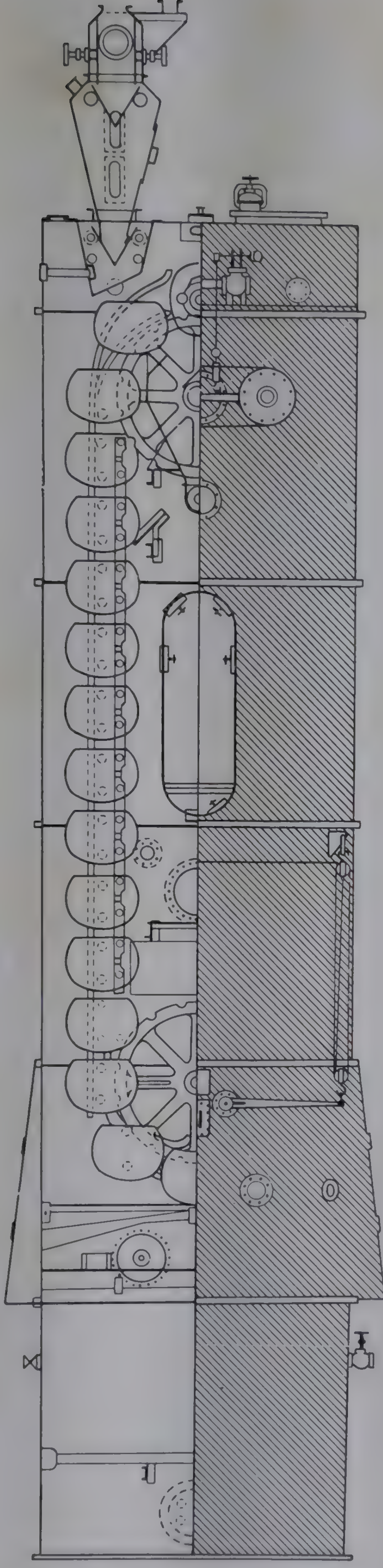

Fig. 17.2. Bollmann extractor

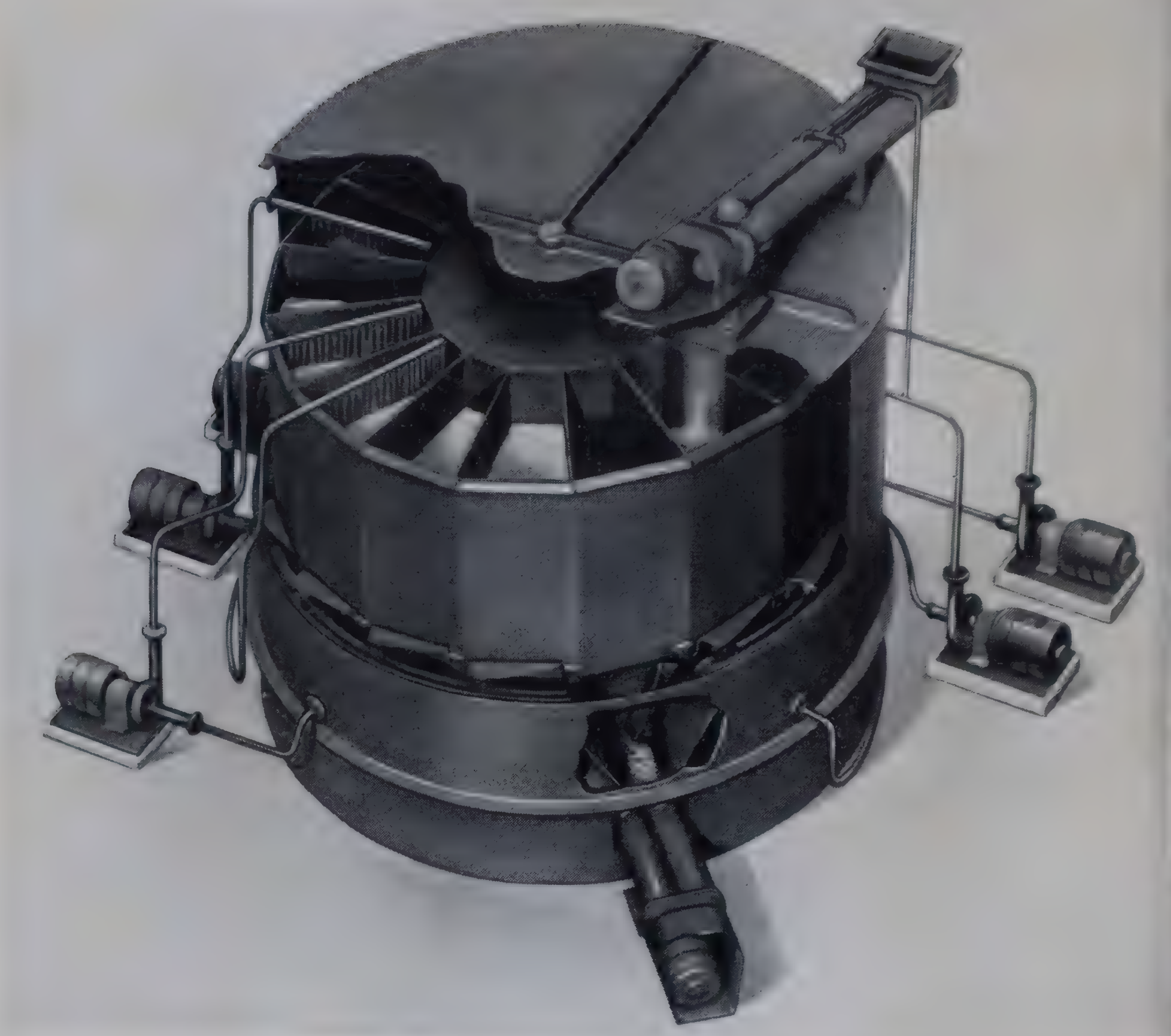

Fig. 17.4. Continuous rotary extractor

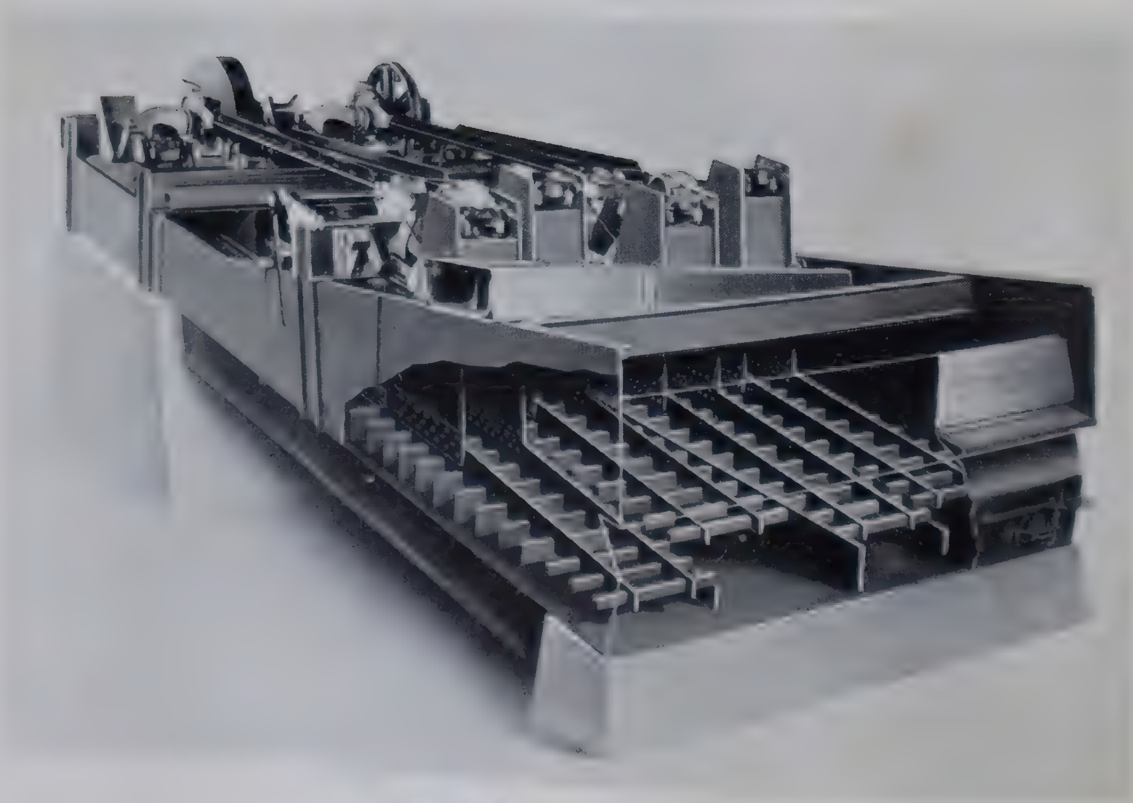

Fig. 17.5. Dorr classifier

with a number of streams of solvent, each successive stream being more dilute than the previous one, and finally with pure solvent before it is discharged.

### Leaching of Coarse Solids

The simple batch plant which is used for coarse solids consists of a cylindrical vessel in which the solids rest on a perforated support. The solvent is sprayed over the solids and, after extraction is complete, the residue is allowed to drain. If the solid contains such a high proportion of solute that it disintegrates, it is treated with solvent in a tank and the solution is decanted.

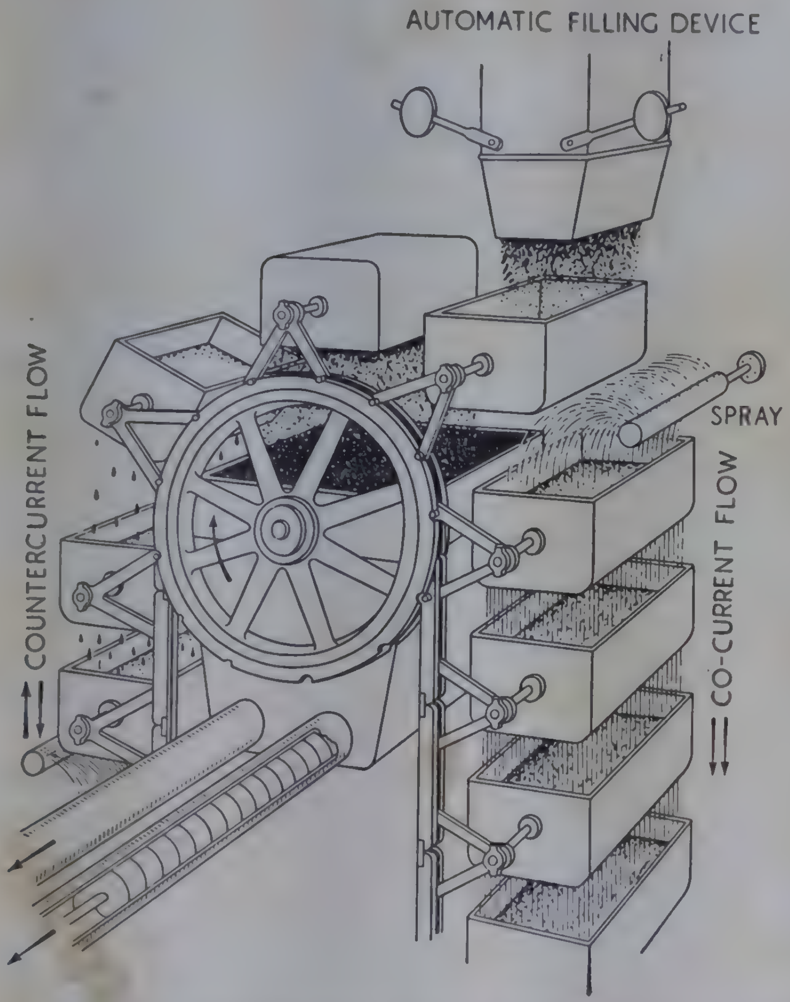

Fig 17.3. Bollmann extractor—filling and emptying of baskets

In the simple countercurrent system, the solid is contained in a number of tanks and the solvent flows through each in turn. The first vessel contains solid which is almost extracted and the last contains fresh solid. After a time, the first tank is disconnected and a fresh charge is introduced at the far end of the battery. The solvent may flow by gravity or be fed by positive pressure, and is generally heated before it enters each tank. The system is unsatisfactory in that it involves frequent interruption while the tanks are replaced, and countercurrent flow is not obtained within the units themselves.

A continuous unit in which countercurrent flow is obtained is the tray classifier, such as the Dorr Classifier (Fig. 17.5). Solid is introduced near the bottom of a sloping tank and is gradually moved up by means of a rake. The solvent enters at the top and flows in the opposite direction to the solid and passes under a baffle before finally being discharged over a weir (Fig. 17.6). The classifier operates satisfactorily provided the solid does not disintegrate, and it can be arranged so that the solids are given ample time to drain before they are discharged. A number of these units can be connected in series to give counter-current flow.

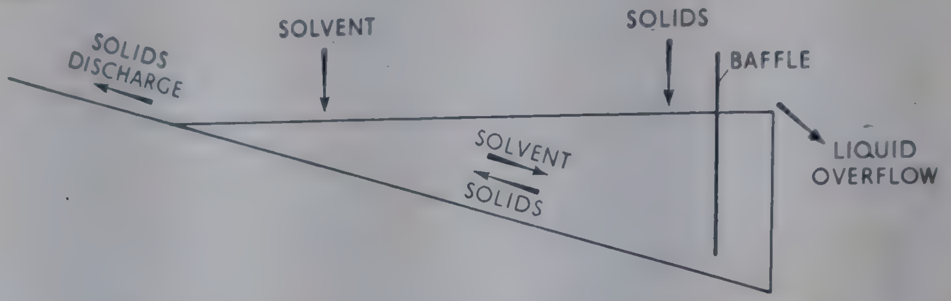
SOLIDS DISCHARGE
SOLVENT
SOLIDS
BAFFLE
SOLVENT
SOLIDS
LIQUID OVERFLOW

Fig. 17.6. Flow of solids and liquids in Dorr classifier

A plant has been successfully developed in Australia for the extraction of potassium sulphate from alums containing about 25% soluble constituents. After roasting, the material is soft and porous with a size range from ½ in. to very fine particles, with 95% greater than 100 mesh, and is leached at 100°C with a solution that is saturated at 30°C, the flow being as shown in Fig. 17.7. The make-up water, which is used for washing the extracted solid, is required to replace that removed in the residue of spent solid, in association with the crystals, and by evaporation in the leaching tank and the crystalliser.

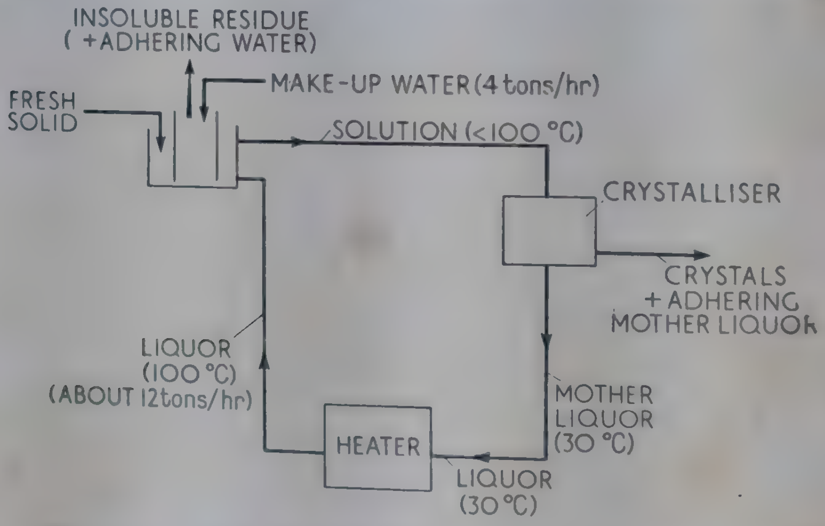
INSOLUBLE RESIDUE ( +ADHERING WATER)
FRESH SOLID
MAKE-UP WATER (4 tons/hr)
SOLUTION (<100 °C)
CRYSTALLISER
CRYSTALS + ADHERING MOTHER LIQUOR
LIQUOR (100 °C) (ABOUT 12tons/hr)
MOTHER LIQUOR (30 °C)
HEATER
LIQUOR (30 °C)

Fig. 17.7. Flow diagram for continuous leaching plant

The leaching plant (Fig. 17.9) consists of an open tank, 10 feet in diameter, into the outer portion of which the solid is continuously introduced from an annular hopper. Inside the tank a six foot diameter vertical pipe rotates very slowly at the rate of about one revolution every 40 minutes. It carries three ploughs stretching to the circumference of the tank, and these gradually force the solid through holes into the inside of the pipe. A hollow shaft, about 3 feet in diameter, rotates in the centre of the tank at about one revolution in 3 minutes and carries a screw conveyor which lifts the solid and finally discharges it

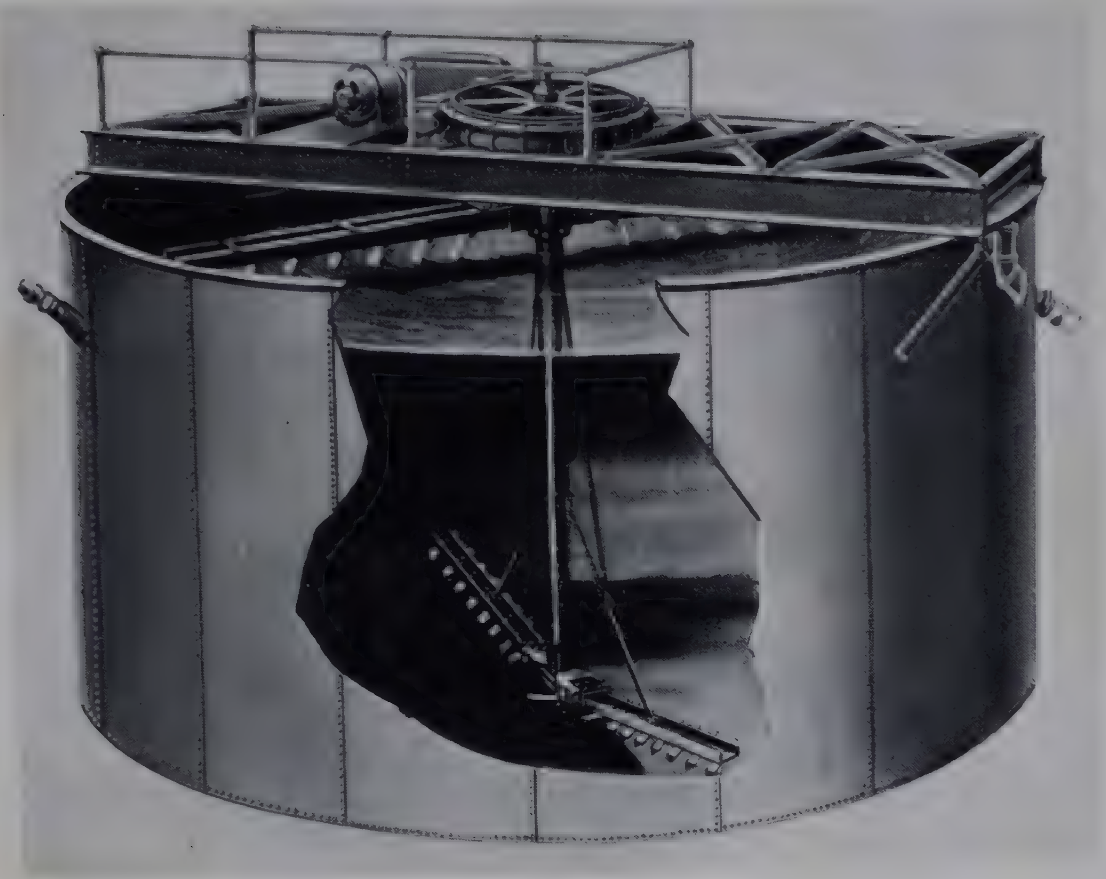

Fig. 17.8. Dorr agitator

through an opening so that it falls down the shaft and is deflected into a waste pipe passing through the bottom of the tank. Leaching takes place in the outer portion of the tank where the reheated mother liquor rises through the descending solid. The make-up water is introduced under the flutes of the screw elevator, flows down over the solid and then joins the reheated mother liquor. Thus countercurrent extraction takes place in the outer part of the tank and countercurrent washing in the central portion. The plant described is said to operate efficiently and gives between 85 and 90% extraction, as compared with only 50% in the batch plant which it replaced.

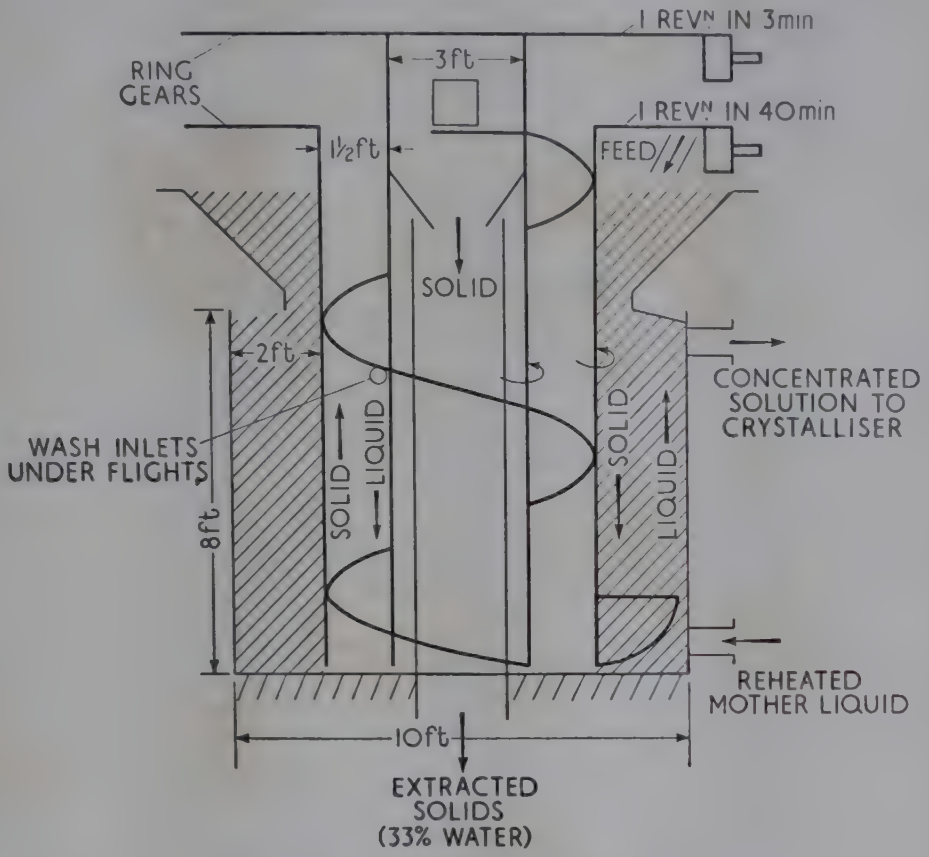
I REVN IN 3min
RING GEARS
3ft
I REVN IN 40min
1½ft
FEED
SOLID
2ft
CONCENTRATED SOLUTION TO CRYSTALLISER
WASH INLETS UNDER FLIGHTS
8ft
SOLID
LIQUID
SOLID
LIQUID
REHEATED MOTHER LIQUID
10ft
EXTRACTED SOLIDS (33% WATER)

Fig. 17.9. Continuous leaching tank

## Leaching of Fine Solids

Whereas coarse solids can be leached by causing the solvent to pass through a bed of the material, fine solids offer too high a resistance to flow. Particles less than about 200 mesh may be maintained in suspension with only a small amount of agitation, and as the total surface area is large an adequate extraction can be effected in a reasonable time. Because of the low settling velocity of the particles and their large surface, the subsequent separation and washing operations are more difficult for fine materials than for coarse solids.

Agitation can be achieved either by the use of mechanical stirrer or by means of compressed air. If a paddle stirrer is used, precautions must be taken to prevent the whole of the liquid being swirled, with very little relative motion occurring between solids and liquid. The stirrer is often placed inside a central tube (Fig. 17.10) and the shape of the blades so arranged that the liquid is lifted upwards through the tube; the liquid then discharges at the top and flows downwards outside the tube, thus giving continuous circulation.

An example of an agitated vessel in which compressed air is used is the Pachuca Tank (Fig. 17.11). This is a cylindrical tank with a conical bottom, fitted with a central pipe connected to an air supply. Continuous circulation is then obtained with the central pipe acting as an air lift. Additional air jets are provided in the conical portion of the base and are used for dislodging any material that settles out.

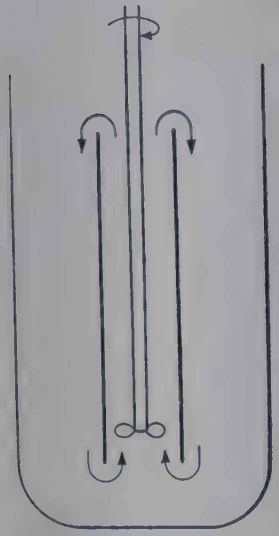

Fig. 17.10. Simple stirred tank

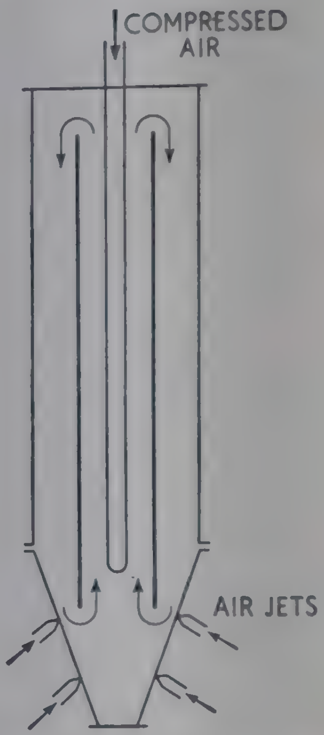

Fig. 17.11. Pachuca tank

The Dorr Agitator (Fig. 17.8), which also uses compressed air for stirring, consists of a cylindrical flat-bottomed tank fitted with a central air lift inside a hollow shaft which slowly rotates. To the bottom of the shaft are fitted rakes which drag the solid material to the centre as it settles, so that it is picked up by the air lift. At the upper end of the shaft the air lift discharges into a perforated launder which distributes the suspension evenly over the surface of the liquid in the vessel. When the shaft is not rotating the rakes automatically fold up so as to prevent the plant from seizing up if it is shut down full of slurry. This type of agitator can be used for batch or continuous operation. In the latter case the entry and delivery points are situated at opposite sides of the tank. The discharge pipe is often in the form of a flexible connection which can be arranged to take off the product from any desired depth. Many of these agitators are heated by steam coils.

If the soluble material dissolves very rapidly, extraction can be carried out in a thickener, such as the Dorr Thickener described in Chapter 15. It gives only slight agitation and automatically separates the solid residue from the clear solution. Thickeners are also extensively used for separating the discharge from an agitator, and a number are frequently connected in series to give countercurrent washing of the residue.

## COUNTERCURRENT WASHING

Where the residual solid after separation is still mixed with an appreciable amount of solution, it is generally desirable to pass it through a battery of washers, arranged to give countercurrent flow of the solids and the solvent as shown in Fig. 17.12. If the solids are relatively coarse a number of classifiers may be used, and with the more usual case of fine solids thickeners are generally employed. In each unit a liquid, referred to as the overflow, and a mixture of insoluble residue and solution, referred to as the underflow, are brought into contact so that intimate mixing is achieved and the solution leaving in the overflow has the same composition as that associated with the solids in the underflow. Each unit then represents an ideal stage. In some cases perfect mixing may not be achieved, and allowance must be made for the lower efficiency of the stage.

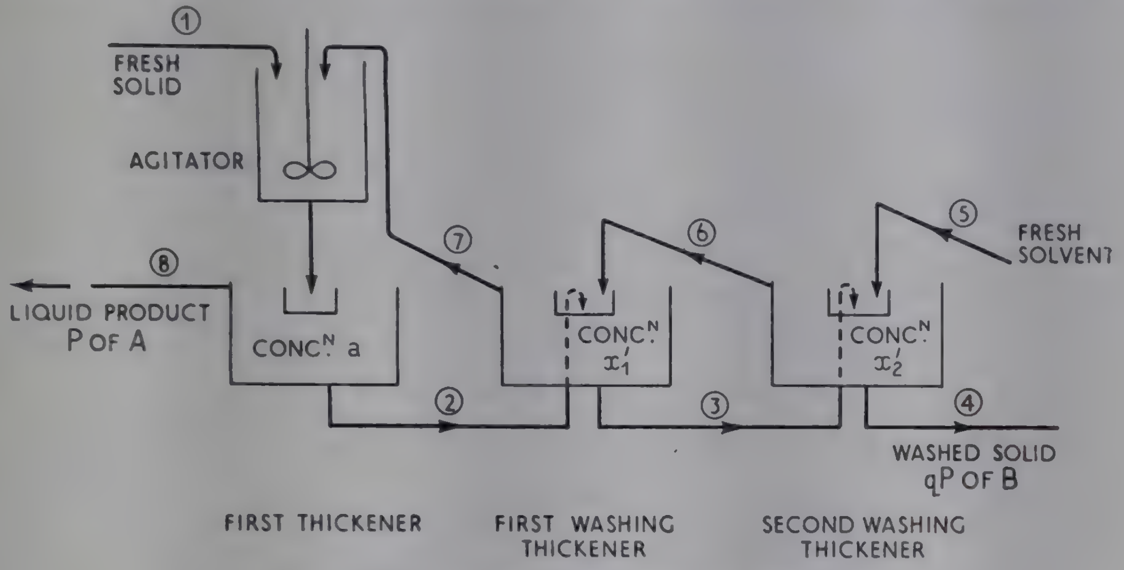

Fig. 17.12. Agitator and washing system

By means of a series of material balances, the compositions of all the streams in the system shown in Fig. 17.12 can be calculated on the assumption that the whole of the solute has been dissolved and that equilibrium is reached in each of the thickeners. In many cases it may only be necessary to determine the compositions of the streams entering and leaving, and the simplified methods given later may then be used.

In order to define such a system completely, the six quantities enumerated below must be specified. The first four relate to the quantities and compositions of the materials used. Item 5 specifies the manner in which each unit operates. Item 6 involves either the number of units or a specification of the duty required of the plant.

(1) The composition of the solvent fed to the system—in particular, the concentration of soluble material already present.

(2) The quantity of solvent used; alternatively the concentration of the solution to be produced may be specified, and the corresponding amount of solvent calculated from a material balance.

(3) The composition of the solid to be leached.

(4) The amount of solid fed to the system; alternatively, the amount of soluble or insoluble material required as product may be specified, and the necessary amount of solid feed then calculated from a material balance.

(5) The amount of liquid discharged with the solid in the underflow from each of the thickeners.

(6) The number and arrangement of the units; the purity of the product from the plant can then be calculated. Alternatively the required purity of the washed solid may be stated, and the number of units can then be calculated.

In the following example, we will consider a solid consisting of a soluble constituent $A$, and an insoluble constituent $B$. Leaching is carried out with a pure solvent $S$ and a solution is produced containing a mass $a$ of $A$, per unit mass of $S$: the total mass of $A$ in solution is to be $P$. It will be assumed that the quantity of solvent removed in the underflow from each of the thickeners is the same, and is independent of the concentration of the solution in that thickener; suppose that unit mass of the insoluble material $B$ removes a mass $s$ of solvent $S$ in association with it. Perfect mixing in each thickener will be assumed and any adsorption of solute on the surface of the insoluble solid will be neglected. In a given thickener therefore, the ratio of solute to solvent will be the same in the underflow as in the overflow.

The compositions of the various streams can be calculated in terms of three unknowns; $x'_1$ and $x'_2$, the ratios of solute to solvent in the first and second washing thickeners respectively, and $qP$, the amount of insoluble solid $B$ in the underflow streams. An overall material balance is first made and then a balance is made on the agitator and its thickener combined, the first washing thickener and the second washing thickener. The procedure is as follows.

For the overall balance, streams 1 and 8 can be recorded immediately. Stream 4 is then obtained from the knowledge that the whole of $B$ appears in the underflow, that the ratio $\frac{S}{B}$ is $s$ and that the ratio $\frac{A}{S}$ is $x'_2$. Stream 5 is obtained by difference. As pure solvent is fed to the system, a relation is obtained by equating the solute content of this stream to zero; this enables $q$ to be eliminated in terms of $x'_2$. For the agitator and separating thickener, streams 1 and 8 have already been determined, and stream 2 is obtained in the same way as stream 4 on the previous balance. Stream 7 is then obtained by differences. It is then possible to proceed in this manner from unit to unit because two streams are always common to consecutive units. Further equations are then obtained from the knowledge that the ratios $\frac{A}{S}$ for the overflows from the two washing thickeners are equal to $x'_1$ and $x'_2$ respectively. Solution of these simultaneous equations for $x'_2$ and $x'_1$ gives each of the streams in terms of known quantities. It will be apparent that this procedure would be laborious if a larger number of units were involved, and therefore alternative methods are used to obtain the compositions of the end streams. The above method would also involve a number of trial and error solutions if it were necessary to calculate the number of units required to give a certain degree of washing. The application of the method will be readily understood from the following example.

**Example.** Caustic soda is manufactured by the lime-soda process by treating a solution of sodium carbonate in water (25 tons per day of $Na_2CO_3$) with the theoretical requirement of lime, and after the reaction is complete the $CaCO_3$ sludge, containing by weight one part of $CaCO_3$ per nine parts of water, is fed continuously to three thickeners in series and is washed countercurrently (Fig. 17.13). Calculate the necessary rate of feed of neutral water to the thickeners so that the calcium carbonate, on drying, contains only 1% of sodium hydroxide. The solid discharged from each thickener contains one part by weight of calcium carbonate to three of water. The concentrated wash liquid is mixed with the contents of the agitator before feeding to the first thickener.

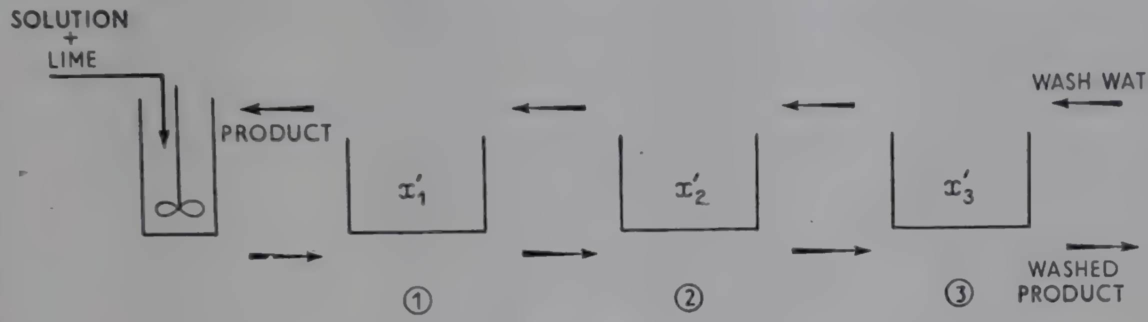

Fig. 17.13.

**Solution**

$$Na_2CO_3 + Ca(OH)_2 = 2NaOH + CaCO_3$$
$$106 \text{ tons} \quad = 80 \text{ tons} + 100 \text{ tons}$$

Let $x'_1$, $x'_2$, $x'_3$ be the solute/solvent ratios in thickeners 1, 2 and 3. The quantities of $CaCO_3$, NaOH and water in each of the streams will be calculated for every 100 tons of calcium carbonate.

| *Overall balance* | $CaCO_3$ | NaOH | Water |
|---|---|---|---|
| Feed from reactor | 100 | 80 | 900 |
| Feed as washwater | — | — | $W_f$ (say) |
| Product-underflow | 100 | $300x'_3$ | 300 |
| Product-overflow | — | $80–300x'_3$ | $600 + W_f$ |
| *Thickener 1* | | | |
| Feed from reactor | 100 | 80 | 900 |
| Feed-overflow | — | $300(x'_1 - x'_3)$ | $W_f$ |
| Product-underflow | 100 | $300x'_1$ | 300 |
| Product-overflow | — | $80–300x'_3$ | $600 + W_f$ |
| *Thickener 2* | | | |
| Feed-underflow | 100 | $300x'_1$ | 300 |
| Feed-overflow | — | $300(x'_2 - x'_3)$ | $W_f$ |
| Product-underflow | 100 | $300x'_2$ | 300 |
| Product-overflow | — | $300(x'_1 - x'_3)$ | $W_f$ |
| *Thickener 3* | | | |
| Feed-underflow | 100 | $300x'_2$ | 300 |
| Feed-water | — | — | $W_f$ |
| Product-underflow | 100 | $300x'_3$ | 300 |
| Product-overflow | — | $300(x'_2 - x'_3)$ | $W_f$ |

Since the final underflow must contain only 1% of NaOH,

$$\frac{300x'_3}{100} = 0{\cdot}01$$

If equilibrium is achieved in each of the thickeners, the ratio of NaOH to water will be the same in the underflow and the overflow.

Thus

$$\frac{300(x'_2 - x'_3)}{W_f} = x'_3$$

$$\frac{300(x'_1 - x'_3)}{W_f} = x'_2$$

$$\frac{80\text{–}300x'_3}{600 + W_f} = x'_1$$

Solution of these four simultaneous equations gives

$$x'_3 = 0{\cdot}0033 \qquad x'_2 = 0{\cdot}0142, \qquad x'_1 = 0{\cdot}05, \qquad W_f = 980$$

Thus the amount of water required for washing 100 tons per day of $CaCO_3$ is 980 tons per day.

Solution fed to reactor contains 25 tons of $Na_2CO_3$ per day. This is equivalent to 23·6 tons per day of $CaCO_3$.

Thus actual feed of water required = 980 × 0·236

= 230 tons per day

### Treatment for Battery of Washing Thickeners

Suppose that, in a battery of thickeners arranged in series for countercurrent washing, the amount of solvent removed with the insoluble solid in the underflow is constant, being independent of the concentration of the solution in the thickener. The amount of solvent leaving each thickener in the underflow will then be the same, and therefore the amount of solvent in the overflow will also be the same. Hence the ratio of the solvent discharged in the overflow to that in the underflow is constant.

$$\text{Let } R = \frac{\text{Amount of solvent discharged in the overflow}}{\text{Amount of solvent discharged in the underflow}} \qquad \ldots.(17.8)$$

If perfect mixing occurs in each of the thickeners and solute is not preferentially adsorbed on the surface of the solid, the concentration of the solution in the overflow will be the same as that in the underflow. If it is then assumed that all the solute has been brought into solution in the agitators,

$$R = \frac{\text{Amount of solute discharged in the overflow}}{\text{Amount of solute discharged in the underflow}} \qquad \ldots.(17.9)$$

$$= \frac{\text{Amount of solution discharged in the overflow}}{\text{Amount of solution discharged in the underflow}} \qquad \ldots.(17.10)$$

It will be noted that these relations apply only to the washing thickeners and not, in general, to the primary thickener in which the product from the agitators is first separated.

Consider a system consisting of *n* washing thickeners arranged for countercurrent washing of a solid from a leaching plant, in which the whole of the soluble material is dissolved. The suspension is separated in a thickener and the underflow from this thickener is fed to the washing system (Fig. 17.14).

Work to a basis of unit mass of insoluble solid.

Let $L_1, \ldots L_h, \ldots L_n$ be the amounts of solute in the overflows from washing thickeners 1 to $n$, respectively.

Let $w_1, \ldots w_h, \ldots w_n$ be the corresponding quantities of solution.

An amount $w_{n+1}$ of wash liquid, fed to the $n$th thickener, contains an amount $L_{n+1}$ of solute.

Let $S_2, \ldots S_{h+1}, \ldots S_{n+1}$ and $W_2, \ldots W_{h+1}, \ldots W_{n+1}$ be the amounts of solute and solution in the underflows from the thickeners.

Let $S_1$ and $W_1$ be the amounts of solute and solution with the solids which are fed to the system for washing. The solvent associated with these solids is taken as the same as that in the underflows from the washing thickeners.

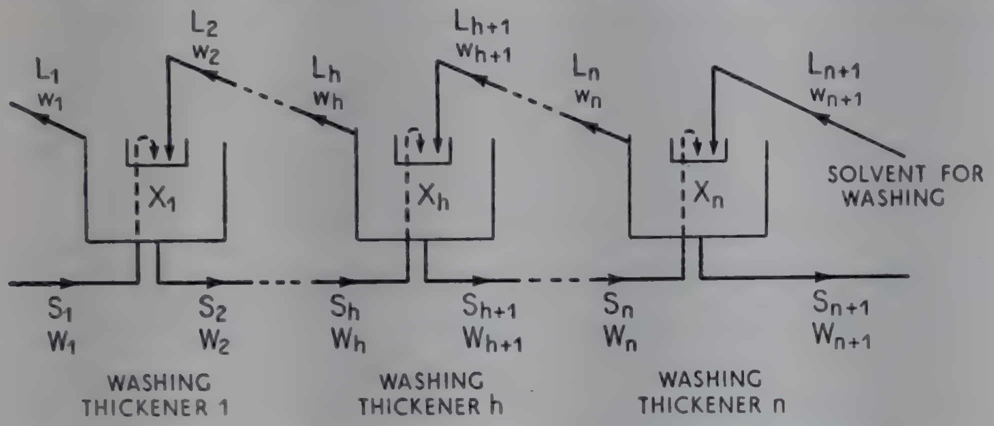
L1
w1
L2
w2
Lh
wh
Lh+1
wh+1
Ln
wn
Ln+1
wn+1
SOLVENT FOR WASHING
X1
Xh
Xn
S1
W1
S2
W2
Sh
Wh
Sh+1
Wh+1
Sn
Wn
Sn+1
Wn+1
WASHING THICKENER 1
WASHING THICKENER h
WASHING THICKENER n

Fig. 17.14. Series of thickeners arranged for countercurrent washing

Now, taking a solute balance on thickener $h$,

$$S_h - S_{h+1} = L_h - L_{h+1} \qquad \ldots.(17.11)$$

Again taking a balance on solution,

$$W_h - W_{h+1} = w_h - w_{h+1} \qquad \ldots.(17.12)$$

Further
$$R = \frac{L_h}{S_{h+1}} = \frac{w_h}{W_{h+1}}$$

i.e.
$$L_h = R \,.\, S_{h+1} \qquad \ldots.(17.13)$$

and
$$w_h = R \,.\, W_{h+1} \qquad \ldots.(17.14)$$

Now taking a balance on solute for each of the thickeners in turn,

Thickener $n$:
$$S_n - S_{n+1} = L_n - L_{n+1} = R \,.\, S_{n+1} - L_{n+1} = R \,.\, S_{n+1} - L_{n+1}$$

Thickener $n - 1$:
$$S_{n-1} - S_n = L_{n-1} - L_n = R \,.\, S_n - R \,.\, S_{n+1} = R^2 \,.\, S_{n+1} - R \,.\, L_{n+1}$$

..........................................................................................

Thickener 2:
$$S_2 - S_3 = L_2 - L_3 = R \,.\, S_3 - R \,.\, S_4 = R^{n-1} \,.\, S_{n+1} - R^{n-2} \,.\, L_{n+1}$$

Thickener 1:
$$S_1 - S_2 = L_1 - L_2 = R \,.\, S_2 - R \,.\, S_3 = R^n \,.\, S_{n+1} - R^{n-1} \,.\, L_{n+1}$$

Then adding over the whole system,

$$S_1 - S_{n+1} = (R + R^2 + \ldots + R^n)S_{n+1} - (1 + R + \ldots + R^{n-1})L_{n+1}$$

i.e.
$$S_1 = \frac{R^{n+1} - 1}{R - 1} S_{n+1} - \frac{R^n - 1}{R - 1} L_{n+1}$$

i.e.
$$(R - 1)S_1 = (R^{n+1} - 1) \,.\, S_{n+1} - (R^n - 1) \,.\, L_{n+1} \qquad \ldots.(17.15)$$

Thus the amount of solute associated with the washed solid can be calculated in terms of the composition of the solid and of the wash liquid fed to the system. In many cases it will be stipulated that the amount of solute associated with the washed solid residue must not exceed a certain value; it is then possible to calculate directly the minimum number of thickeners necessary in order to achieve this.

If the liquid fed to the washing system is pure solvent, $L_{n+1}$ will be equal to zero and therefore,

$$\frac{S_{n+1}}{S_1} = \frac{R - 1}{R^{n+1} - 1} \qquad \ldots.(17.16)$$

$S_{n+1}/S_1$ represents the fraction of the solute fed to the washing system which remains associated with the washed solids. If in a given case it is specified that this fraction should not exceed a value $f$, the minimum number of washing thickeners required is given by,

$$f = \frac{R - 1}{R^{n+1} - 1}$$

$$\therefore \qquad R^{n+1} = 1 + (R - 1) \,.\, \frac{1}{f}$$

$$\therefore \qquad (n + 1) \log R = \log \left\{ 1 + (R - 1) \,.\, \frac{1}{f} \right\}$$

$$\therefore \qquad n = \frac{\log \left\{ 1 + (R - 1) \,.\, \frac{1}{f} \right\}}{\log R} - 1 \qquad \ldots.(17.17)$$

$n$ will, in general, not work out to be a whole number. The required number of thickeners will be given by the next higher whole number.

It is sometimes more convenient to work in terms of the total amount of *solution* entering and leaving each thickener.

Now
$$W_h - W_{h+1} = w_h - w_{h+1} \qquad \ldots.(17.12)$$

and
$$w_h = R \,.\, W_{h+1} \qquad \ldots.(17.14)$$

Then, using the same method as before, we have,

$$(R - 1) \,.\, W_1 = (R^{n+1} - 1) \,.\, W_{n+1} - (R^n - 1) \,.\, w_{n+1} \qquad \ldots.(17.18)$$

## Washing in Batch Process

The solid residue obtained from a batch leaching process may be washed by mixing it in a vat with liquid, allowing the mixture to settle, and then decanting the solution. This process can then be repeated until the solid is adequately washed. Let us suppose that, in each decantation operation, the ratio $R'$ of the amount of solvent decanted, to that remaining in association with the insoluble solid, is a constant and independent of the concentration of the solution in the vat.

Then, after the first washing, the fraction of the soluble material remaining behind with the solid in the vat is $\dfrac{1}{R'+1}$. After the second washing, a fraction $\dfrac{1}{R'+1}$ of this remains behind, i.e. $\dfrac{1}{(R'+1)^2}$ of the solute originally present is retained. Similarly after $m$ washing operations, the fraction of the solute retained by the insoluble residue is $\dfrac{1}{(R'+1)^m}$.

## Washing System with Variable Underflow$^{(5)}$

In the systems so far considered, the quantity of solvent, or of solution, removed in association with the insoluble solids has been assumed to be constant and independent of the concentration of solution in the thickener. Consideration will now be given to a similar countercurrent system in which the amount of solvent or solution in the underflow is a function of the concentration of the solution. This treatment is equally applicable to the washing thickeners alone or to the whole system involving agitator and thickeners.

The same notation will be employed as that used for the previous problem (see Fig. 17.14), i.e.,

$L$ and $w$ will denote solute and solution, respectively, in the liquid overflows and

$S$ and $W$ will denote solute and solution in the underflows.

The concentration of the solution in each thickener, defined as the ratio of solute to solution, will be denoted by the symbol $X$. Then considering the overflow from thickener $h$,

$$X_h = \frac{L_h}{w_h} \qquad \ldots .(17.19)$$

and for the underflow,

$$X_h = \frac{S_{h+1}}{W_{h+1}} \qquad \ldots .(17.20)$$

It has already been stated (page 579) that in order to define the system, it is necessary to specify the following quantities or other quantities from which they can be calculated by a material balance.

(1) The composition of the liquid used for washing, i.e. $X_{n+1}$.

(2) The quantity of wash liquid employed, i.e. $w_{n+1}$. Thus $L_{n+1}$ can be calculated from the relation $L_{n+1} = w_{n+1} \cdot X_{n+1}$.

(3) The composition of the solid to be washed, i.e. $S_1$ and $W_1$.

(4) The quantity of insoluble solid to be washed; this is taken as unity.

(5) The quantity of solution removed by the solid in the underflow from the thickeners; this will vary according to the concentration of the solution in the thickener, and it is therefore necessary to know the relation between $W_{h+1}$ and $X_h$. This must be determined experimentally under conditions similar to those under which the plant will operate. The data for $W_{h+1}$ should then be plotted against $X_{h+1}$. On the same graph it is convenient to plot values calculated for $S_{h+1}$ ($= W_{h+1} \cdot X_h$).

(6) The required purity of the washed solid, i.e. $S_{n+1}$. This automatically defines $X_n$ and $W_{n+1}$ whose values can be read off from the graph referred to under (5). Alternatively, the number of thickeners in the washing system may be given, and a calculation made of the purity of the product required. This problem is slightly more complicated and it will therefore be dealt with subsequently.

The solution of the problem depends on the application of material balances with respect to solute and to solution, first over the system as a whole and then over the first $h$ thickeners.

*Balance on the System as a Whole*

Solute: $L_{n+1} + S_1 = L_1 + S_{n+1}$

Thus the solute in the liquid overflow from the system as a whole is given by,

$$L_1 = L_{n+1} + S_1 - S_{n+1} \qquad \text{....(17.21)}$$

Solution: $w_{n+1} + W_1 = w_1 + W_{n+1}$

Thus the solution discharged in the liquid overflow is given by,

$$w_1 = w_{n+1} + W_1 - W_{n+1} \qquad \text{....(17.22)}$$

Then the concentration of the solution discharged from the system is obtained by substituting from equations 17.21 and 17.22 in equation 17.19

i.e.

$$X_1 = \frac{L_{n+1} + S_1 - S_{n+1}}{w_{n+1} + W_1 - W_{n+1}} \qquad \text{....(17.23)}$$

*Balance on the first h Thickeners*

Solute: $L_{h+1} + S_1 = L_1 + S_{h+1}$

Thus the amount of solute in the liquid fed to thickener $h$

$$\begin{aligned} &= L_{h+1} = L_1 + S_{h+1} - S_1 \\ &\qquad = L_{n+1} - S_{n+1} + S_{h+1} \text{ (from equation 17.21)} \end{aligned} \qquad \text{....(17.24)}$$

Solution: $w_{h+1} + W_1 = w_1 + W_{h+1}$

Thus the amount of solution fed to thickener $h$

$$\begin{aligned} &= w_{h+1} = w_1 + W_{h+1} - W_1 \\ &\qquad = w_{n+1} - W_{n+1} + W_{h+1} \text{ (from equation 17.22)} \end{aligned} \qquad \text{....(17.25)}$$

Thus the concentration of the solution fed to thickener $h$ is given by substituting from equations 17.24 and 17.25 in equation 17.19.

i.e.
$$X_{h+1} = \frac{L_{n+1} - S_{n+1} + S_{h+1}}{w_{n+1} - W_{n+1} + W_{h+1}} \quad \ldots.(17.26)$$

In equation 17.23, all the quantities except $X_1$ are known, and therefore $X_1$ can be calculated. [It should be noted here that if, instead of the quantity of wash liquid fed to the system, the concentration of the solution leaving the system had been given, equation 17.23 could have been used to calculate $w_{n+1}$.] Now that $X_1$ has been evaluated, the solution of the problem depends on the application of equation 17.26 in successive stages. The only unknown quantities in equation 17.26 are $X_{h+1}$, $S_{h+1}$ and $W_{h+1}$.

Applying equation 17.26 to the first stage ($h = 1$), we have

$$X_2 = \frac{L_{n+1} - S_{n+1} + S_2}{w_{n+1} - W_{n+1} + W_2} \quad \ldots.(17.27)$$

Since $X_1$ is now known, the values of $S_2$ and $W_2$ can be read off from the graph, in which $S_{h+1}$ and $W_{h+1}$ are plotted against $X_h$. After substitution of these values in the equation, $X_2$ can be calculated. The next step is to apply equation 17.26 for $h = 2$. $X_2$ is now known so that $S_3$ and $W_3$ can be read off from the graph, and the value of $X_3$ can then be calculated. It is thus possible to apply equation 17.26 in this way in successive stages until the value obtained for $S_{h+1}$ is brought down to the specified value of $S_{n+1}$. The number of washing thickeners required to reduce the solute associated with the washed solid to a specified figure is thus readily calculated. In general, of course, it will not be possible to choose the number of thickeners so that $S_{h+1}$ is exactly equal to $S_{n+1}$.

It will be seen that the purity of the washed solid must be known before equations 17.23 and 17.26 can be applied. If in a given problem it is necessary to calculate the degree of washing obtained by the use of a certain number of washing thickeners, an initial assumption of the values of $S_{n+1}$ and $W_{n+1}$ must be made before the problem can be tackled. As a first step we can take an average value for $R$ and calculate $S_{n+1}$ from equation 17.16. The method, as already given, should then be applied for the number of thickeners specified in the problem, and the calculated and assumed values of $S_{n+1}$ compared. If the calculated value is higher than the assumed value, the latter is too low. The calculated values of $S_{n+1}$ can then be plotted against the corresponding assumed values. The correct solution is then denoted by the point at which the two values agree.

**Example.** A plant produces 100 tons per day of titanium dioxide pigment which must be 99·9% pure when dried. The pigment is produced by precipitation and the material, as prepared, is contaminated with 1 ton of salt solution, containing 0·55 tons of salt, per ton of pigment. The material is washed countercurrently with water in a number of thickeners arranged in series. How many thickeners will be required if water is added at the rate of 200 tons per day and the solid discharged from each thickener removes ½ ton of solvent per ton of pigment?

What will be the required number of thickeners if the amount of solution removed in association with the pigment varies in the following way with the concentration of the solution in the thickener?

| Concentration (solute/solution) | Solution/unit mass of pigment |
|---|---|
| 0 | 0·30 |
| 0·1 | 0·32 |
| 0·2 | 0·34 |
| 0·3 | 0·36 |
| 0·4 | 0·38 |
| 0·5 | 0·40 |

The concentrated wash liquor is mixed with the material fed to the first thickener.

**Solution**

*Part* 1

Overall balance on plant in tons per day

| | $TiO_2$ | Salt | Water |
|---|---|---|---|
| Feed from reactor | 100 | 55 | 45 |
| Wash liquor added | — | — | 200 |
| Washed solid | 100 | 0·1 | 50 |
| Liquid product | — | 54·9 | 195 |

Solvent in underflow from final washing thickener = 50.

Solvent in overflow will be the same as that supplied for washing, i.e. = 200.

$$\frac{\text{Solvent discharged in overflow}}{\text{Solvent discharged in underflow}} = 4 \quad \text{for the washing thickeners.}$$

Liquid product from plant contains 54·9 tons of salt in 195 tons of solvent.

This ratio will be the same in the underflow from the first thickener.

Thus the material fed to the washing thickeners consists of 100 tons $TiO_2$, 50 tons solvent and 50 × 54·9/195 = 14 tons salt.

Required number of thickeners for washing is given by,

$$\frac{4 - 1}{4^{n+1} - 1} = \frac{0{\cdot}1}{14}$$

i.e.

$$4^{n+1} = 421$$

giving

$$4 < n + 1 < 5$$

Thus 4 washing thickeners or a total of *5 thickeners* are required.

*Part* 2

The same symbols will be used as in the text.

By inspection of the data, it is seen that $W_{h+1} = 0{\cdot}30 + 0{\cdot}2X_h$.

Then $$S_{h+1} = W_{h+1} \,.\, X_h = 0{\cdot}30 \,.\, X_h + 0{\cdot}2 \,.\, X_h^2 = 5W_{h+1}^2 - 1{\cdot}5W_{h+1}$$

Consider the passage of unit quantity of $TiO_2$ through the plant.

$$L_{n+1} = 0, \qquad w_{n+1} = 2, \qquad X_{n+1} = 0$$

since 200 tons/day pure solvent are used.

$$S_{n+1} = 0{\cdot}001 \quad \text{and therefore} \quad W_{n+1} = 0{\cdot}3007.$$

$$S_1 = 0{\cdot}55 \quad \text{and} \quad W_1 = 1{\cdot}00$$

Thus the concentration in the first thickener is given by equation 17.23.

$$X_1 = \frac{L_{n+1} + S_1 - S_{n+1}}{w_{n+1} + W_1 - W_{n+1}} = \frac{0 + 0{\cdot}55 - 0{\cdot}001}{2 + 1 - 0{\cdot}3007} = \frac{0{\cdot}549}{2{\cdot}700} = 0.203$$

From equation 17.26,

$$X_{h+1} = \frac{L_{n+1} - S_{n+1} + S_{h+1}}{w_{n+1} - W_{n+1} + W_{h+1}} = \frac{0 - 0{\cdot}001 + S_{h+1}}{2 - 0{\cdot}3007 + W_{h+1}} = \frac{-0{\cdot}001 + S_{h+1}}{1{\cdot}7 + W_{h+1}}$$

Since, $X_1 = 0{\cdot}203$, $W_2 = 0{\cdot}30 + 0{\cdot}2 \times 0{\cdot}203 = 0{\cdot}3406$

and $S_2 = 0{\cdot}3406 \times 0{\cdot}203 = 0{\cdot}0691$

Thus $$X_2 = \frac{0{\cdot}0691 - 0{\cdot}001}{1{\cdot}7 + 0{\cdot}3406} = \frac{0{\cdot}0681}{2{\cdot}0406} = 0{\cdot}0334$$

Since $X_2 = 0{\cdot}0334$, $W_3 = 0{\cdot}30 + 0{\cdot}2 \times 0{\cdot}0334 = 0{\cdot}30668$

and $S_3 = 0{\cdot}3067 \times 0{\cdot}0334 = 0{\cdot}01025$

Thus $$X_3 = \frac{0{\cdot}01025 - 0{\cdot}001}{1{\cdot}7 + 0{\cdot}3067} = \frac{0{\cdot}00925}{2{\cdot}067} = 0{\cdot}00447$$

Since $X_3 = 0{\cdot}00447$, $W_4 = 0{\cdot}30089$ and $S_4 = 0{\cdot}0013$
Hence, by the same method, $X_4 = 0{\cdot}000150$
Since $X_4 = 0{\cdot}000150$, $W_5 = 0{\cdot}30003$ and $S_5 = 0{\cdot}000045$
Thus $S_5$ is less than $S_{n+1}$ and therefore 4 thickeners are required.

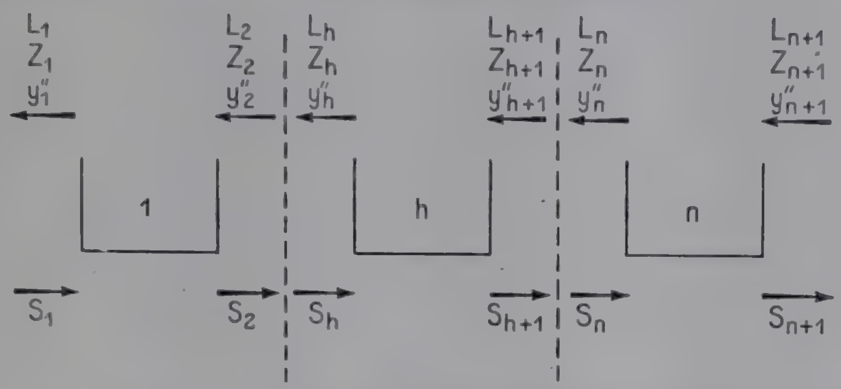

Fig. 17.15. Countercurrent washing system

## Graphical Methods

It is sometimes convenient to use graphical constructions for the solution of countercurrent leaching or washing problems. This may be done by a method similar to the McCabe-Thiele method for distillation which is discussed in Chapter 18, with the overflow and underflow streams corresponding to the vapour and liquid respectively. The basis of this method will now be given, but a generally more convenient method involves the use of triangular diagrams which will be discussed later in some detail.

For the countercurrent washing system shown in Fig. 17.15, the ratio of solute to solvent in the overflow at any stage $y''_h$ can be related to the ratio of solute to insoluble solid in the underflow $S_h$ by means of a simple material balance. Using this notation

$$y''_h = \frac{L_h}{w_h - L_h} = \frac{L_h}{Z_h} \qquad \ldots.(17.28)$$

where $Z_h$ is the amount of solvent in the overflow per unit mass of insoluble solid in the underflow. If the solvent in the underflow is constant throughout the system, $Z$ will not be a function of concentration. Dropping the suffix of $Z$ therefore, and taking a balance on solute over thickeners $h$ to $n$ inclusive,

$$Z(y''_h - y''_{n+1}) = S_h - S_{n+1}$$

i.e. $$y''_h = \frac{S_h}{Z} - \frac{S_{n+1}}{Z} + y''_{n+1} \qquad \ldots.(17.29)$$

Thus a linear relation exists between $y''_h$ and $S_h$.

If the ratio of solvent in the overflow to solvent in the underflow from any thickener is equal to $R$,

$$\frac{L_h}{S_{h+1}} = \frac{w_h - L_h}{W_{h+1} - S_{h+1}} = \frac{Zy_h''}{S_{h+1}} = R$$

i.e.

$$y_h'' = \frac{R}{Z} S_{h+1} \qquad \ldots .(17.30)$$

This is a straight line of slope $\frac{R}{Z}$, and passing through the origin. Equations 17.29 and 17.30 can be represented on a $y'' - S$ diagram, as shown in Fig. 17.16. If pure solvent is used for washing, $y_{n+1}'' = 0$, and the intercept on the $S$ axis is $S_{n+1}$. As the two lines represent the relation between $y_h''$ and both $S_h$ and $S_{h+1}$, the change in composition of the underflow and overflow streams can be determined by a series of stepwise constructions, the number of steps required to change the composition of the overflow from $y_{n+1}''$ to $y_1''$ being the number of stages required.

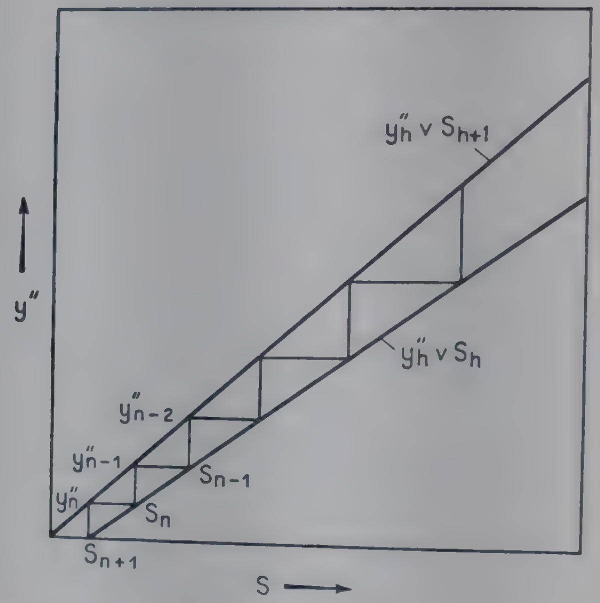

Fig. 17.16. Graphical construction for number of thickeners

For a variable underflow the relation between $y_h''$ and $S_{h+1}$ must be determined experimentally as the two curves are no longer straight lines, but the procedure is similar once they have been drawn. Further, it is assumed that each thickener represents an ideal stage and that the ratio of solute to solvent is the same in the overflow and the underflow. If each stage is only 80% efficient, say, equation 17.30 is no longer applicable, but the same method can be used except that each of the vertical steps will extend only 80% of the way to the curve of $y_h''$ versus $S_{h+1}$.

Further use of graphical methods is discussed by SCHEIBEL(9) but we will confine our attention now to the use of right-angled triangular diagrams.

## THE USE OF RIGHT-ANGLED TRIANGULAR DIAGRAMS⁽¹⁾

Suppose that a total mass $F$ of material is fed to a thickener and is separated into a mass $w'$ of overflow and a mass $W'$ of underflow: it will be assumed that the whole of the insoluble solid appears in the underflow. A material balance then gives that

$$F = W' + w' \qquad \text{....(17.31)}$$

Let $z$, $y$ and $x$ be the fractional compositions of $F$, $w'$ and $W'$, respectively, with respect to any one component in the mixture; the solute $A$, the insoluble solid $B$, or the solvent $S$. Then, taking a material balance on any component,

$$F \,.\, z = W' \,.\, x + w' \,.\, y$$

i.e.

$$(W' + w') \,.\, z = W' \,.\, x + w' \,.\, y \qquad \text{....(17.32)}$$

and

$$z = \frac{W'x + w'y}{W' + w'} \qquad \text{....(17.33)}$$

Then for the solute and solvent respectively we can write,

$$z_A = \frac{W'x_A + w'y_A}{W' + w'} \qquad \text{....(17.33a)}$$

$$z_S = \frac{W'x_S + w'y_S}{W' + w'} \qquad \text{....(17.33b)}$$

The composition of the three-component mixture can be represented on a right-angle triangular diagram. The proportion of solute $A$ in the mixture is plotted as the abscissa, and the proportion of solvent as the ordinate; the proportion of insoluble solid is then obtained by difference. Let the point $a$, $(z_A, z_S)$, represent the composition of the material fed to the thickener, point $b$, $(x_A, x_S)$, the composition of the underflow and point $c$, $(y_A, y_S,)$ the composition of the overflow (Fig. 17.17).

The slope of the line $ab$

$$= \frac{z_S - x_S}{z_A - x_A} = \frac{\dfrac{W'x_S + w'y_S}{W' + w'} - x_S}{\dfrac{W'x_A + w'y_A}{W' + w'} - x_A} = \frac{w'y_S - w'x_S}{w'y_A - w'x_A} = \frac{y_S - x_S}{y_A - x_A}$$

$$\text{....(17.34)}$$

But the slope of the line $bc$ is also $\dfrac{y_S - x_S}{y_A - x_A,}$ so that $a$, $b$ and $c$ lie on the same straight line. Thus, if two streams are mixed, the composition of the mixture will be given by some point on the line joining the points representing the compositions of the constituent streams. Similarly, if one stream is subtracted from another, the composition of the resulting stream will lie at some point on the corresponding straight line produced. The location of the point will depend on the relative quantities in the two streams.

Now
$$(W' + w') . z = W' . x + w' . y \qquad \ldots .(17.32)$$

$$w'(z - y) = W'(x - z)$$

$$\therefore \quad \frac{w'}{W'} = \frac{x - z}{z - y} = \frac{x_A - z_A}{z_A - y_A} = \frac{x_S - z_S}{z_S - y_S} \qquad \ldots .(17.35)$$

Thus the point representing the mixture, divides the line *bc* so that $\frac{ba}{ac} = \frac{w'}{W'}$; that is to say that *a* is nearer to the point corresponding to the larger stream.

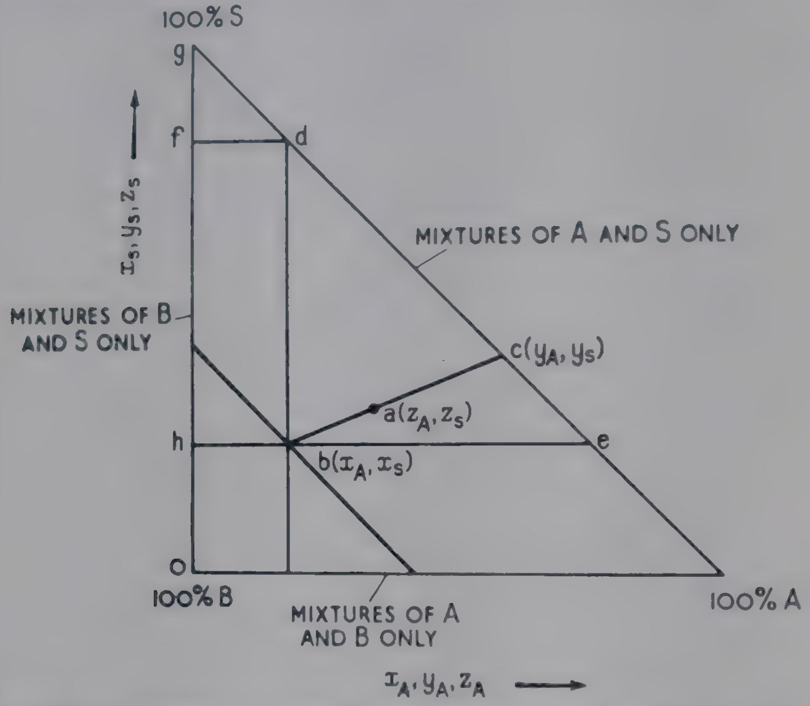

Fig. 17.17. Representation of three-component system on triangular diagram

The proportion of insoluble solid in the underflow, for example, is given by the relation,

$$x_A + x_S + x_B = 1$$

$$\therefore \qquad x_S = -x_A + (1 - x_B) \qquad \ldots .(17.36)$$

Lines representing constant values of $x_B$ are therefore straight lines of slope $-1$; i.e. they are parallel to the hypotenuse of the right angle triangle; the intercept on either axis is $(1 - x_B)$. Thus the hypotenuse represents mixtures containing no insoluble solid, and therefore the compositions of all possible overflows are represented by the hypotenuse. Further, it can be seen from the geometry of the diagram that

$$fd = fg = x_A$$

and
$$Oh = x_S.$$

Thus
$$fh = bd = be$$
$$= 1 - x_A - x_S = x_B.$$

The proportion of the third constituent, the insoluble solid *B*, is therefore given by the distance of the point from the hypotenuse, measured in a direction parallel to either of the main axes.

Points which lie within the triangle represent the compositions of real mixtures of the three components. Each vertex represents a pure component and each of the sides represents a two-component mixture. If two streams are mixed, the composition of the resultant stream is readily obtained and is represented by an addition point which must lie within the diagram. The composition of the material resulting from mixing a number of streams can be obtained by combining streams two at a time. If one stream is subtracted from another, a similar procedure is adopted to determine the composition of the remaining stream. The point so obtained is known as a difference point and must lie on the extension of the line joining the two given points, on the side nearer the one representing the mixed stream.

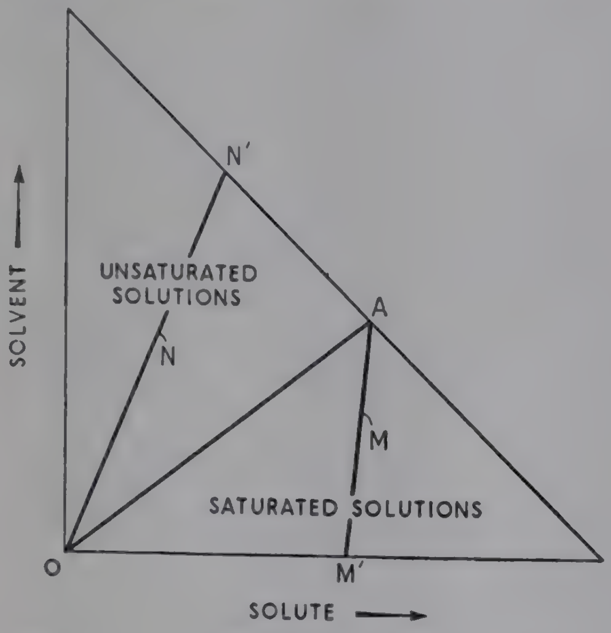

Fig. 17.18. Effect of saturation—solid solute

If we try to abstract from a stream more of a given component than is actually present, the composition of the resulting stream will be imaginary and will be represented by some point outside the triangle. The concept of an imaginary difference point is useful in its application to countercurrent flow processes.

### Effect of Saturation

When the solute is initially present as a solid, the amount that can be dissolved in a given amount of solvent is limited by the solubility of the material. A saturated solution of the solute will be represented by some point, such as *A*, on the hypotenuse of the triangular diagram (Fig. 17.18). The line *OA* represents the compositions of all possible mixtures of saturated solution with insoluble solid, since $x_S/x_A$ is constant at all points on this line. The part of the triangle above *OA* therefore represents unsaturated solutions mixed with the insoluble solid *B*. If a mixture, represented by some point *N*, is separated into solid and liquid, it will yield a solid, of composition represented by *O*, i.e. pure component *B*, and an unsaturated solution (*N′*). Again, the lower part of the diagram represents mixtures of insoluble solid, undissolved solute, and saturated solution. Thus, if a mixture *M* is separated into a liquid and solid fraction, it will yield a liquid, indicated by point *A*, (i.e. a saturated solution) and a solid, consisting of *B* together with undissolved *A* (*M*′).

If the solute is initially in a liquid form and the solvent is completely miscible with it, the whole of the triangle will represent unsaturated conditions. If the solvent and the solute are not completely miscible, the area can be divided into three distinct regions, as shown in Fig. 17.19. In region 1, the solvent is present as an unsaturated solution in the solute. In region 2, the liquid consists of two phases; a saturated solution of *A* in *S* and a saturated solution of *S* in *A*, in various proportions. In region 3, the liquid consists of an unsaturated solution of solute in solvent.

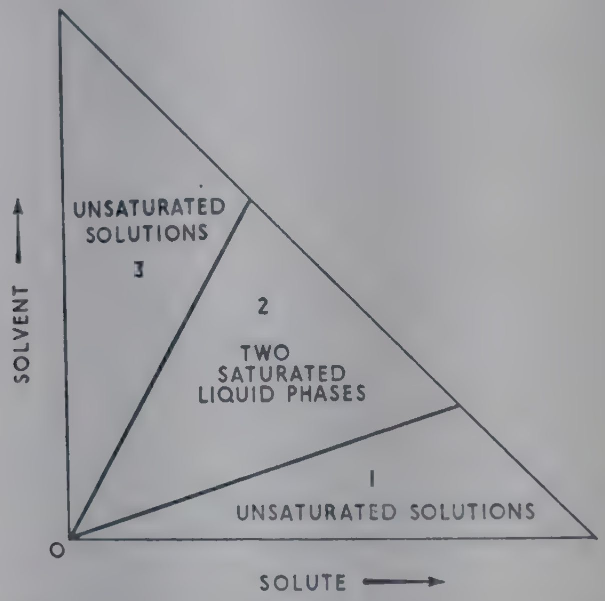

Fig. 17.19. Effect of saturation—liquid solute

In any plant, in which solvent is used to wash the adhering film of solution from the surface of the insoluble solid, the solution formed as a result of mixing in any of the units will consist of unsaturated solution. In an extracting plant, sufficient solvent will normally be added in order to dissolve the solute completely, so that, in practice, the solutions considered will rarely reach saturation.

### Underflow

The conditions applying in an industrial thickener can be represented by means of the triangular diagram. In any equilibrium stage, the quantity of solution in the underflow may be a function of the concentration of the solution in the thickener, and the concentration of the overflow solution will be the same as that in the underflow. Let the curved line *EF* (Fig. 17.20) represent the experimentally determined composition of the underflow for various concentrations. Any point *f* on this line represents the composition of a mixture of pure *B* with a solution of composition *g*, and *Of*/*fg* is the ratio of solution to solids in the underflow. If the amount of solution removed in the underflow is not affected by its concentration, the fractional composition of the underflow with respect to the insoluble material *B* ($x_B$) is a constant, and is represented by a straight line, through *E*, parallel to the hypotenuse. Let *EF*′ represent such a line. Point $E$ represents the composition of the underflow when the solution is infinitely weak, i.e. when it consists of pure solvent. If $K$ is the mass of solution removed in the underflow per unit mass of solids, the ordinate of $E$ is given by,

$$x_S = \frac{K}{K+1}$$

The equation of the line $EF'$ is therefore,

$$x_S = -x_A + \frac{K}{K+1}$$

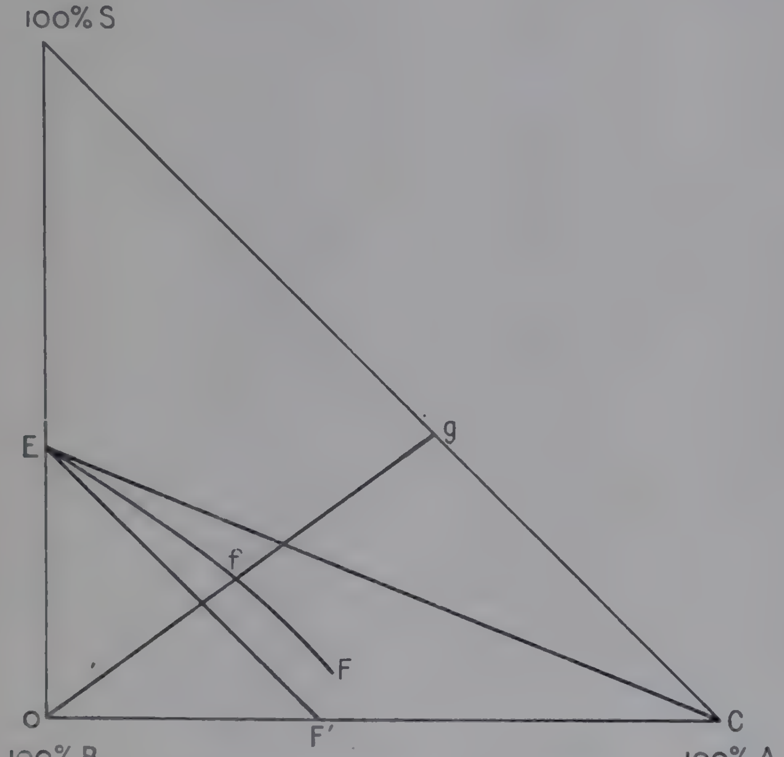

Fig. 17.20. Representation of underflow stream

If the ratio of solvent to insoluble solid is constant (and equal to $s$) in the underflow, however, the line $EF$ will be a straight line passing through the vertex $C$ of the triangle, which corresponds to pure solute $A$. $E$ is then given by the co-ordinates,

$$x_A = 0 \qquad x_S = \frac{s}{s+1}$$

### Countercurrent Systems

Consider a countercurrent system consisting of $n$ thickeners, as shown in Fig. 17.21. The net flow to the right must necessarily be constant throughout the system, if no material enters or leaves at intermediate points.

$$\text{Net flow to the right} = F' = W'_h - w'_h, \text{ etc.} \qquad \ldots.(17.37)$$

The point representing the stream $F'$ will be a difference point, since it will represent the composition of the stream which must be added to $w'_h$ to give

$W'_h$. Now, in general, in a countercurrent flow system of this sort, the net flow of all the constituents will not be in the same direction. Thus one or more of the fractional compositions in this difference stream will be negative and the

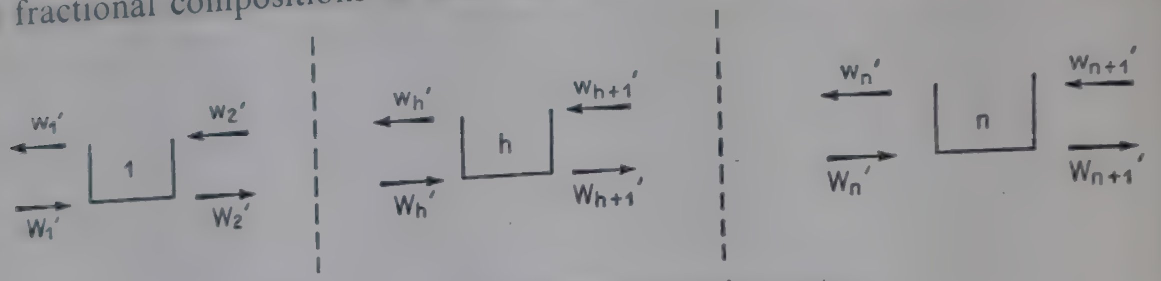
w1'
W2'
1
W1'
W2'
wh'
wh+1'
h
Wh'
Wh+1'
wn'
wn+1'
n
Wn'
Wn+1'

Fig. 17.21. Countercurrent extraction system

difference point will lie outside the triangle. A balance on the whole system, as shown in Fig. 17.22, gives

$$W'_1 - w'_1 = W'_{n+1} - w'_{n+1}$$

and $$W'_1 x_1 - w'_1 y_1 = W'_{n+1} \cdot x_{n+1} - w'_{n+1} \cdot y_{n+1},$$

for any of the three components.

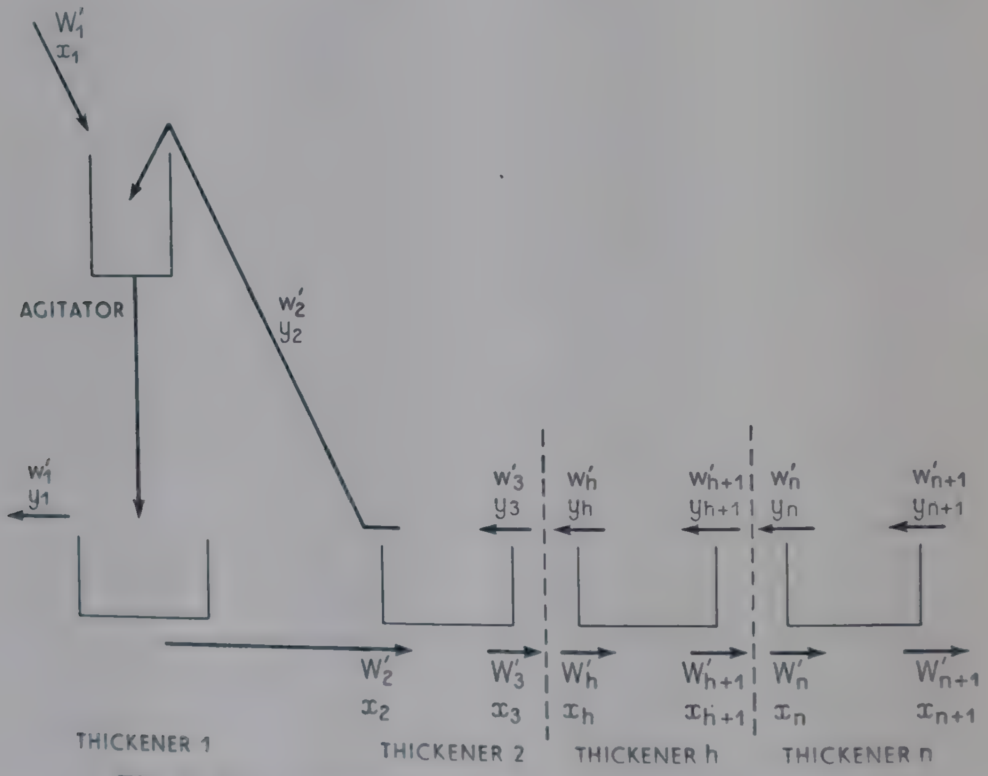
W'1
x1
AGITATOR
w'2
y2
w'1
y1
w'3
y3
w'h
yh
w'h+1
yh+1
w'n
yn
w'n+1
yn+1
W'2
x2
W'3
x3
W'h
xh
W'h+1
xh+1
W'n
xn
W'n+1
xn+1
THICKENER 1
THICKENER 2
THICKENER h
THICKENER n

Fig. 17.22. Countercurrent washing system and agitator

The total net flow of material to the right at some intermediate point

$$= W'_h - w'_h,$$

and the net flow of one of the constituents

$$= W'_h \cdot x_h - w'_h \cdot y_h$$

The fractional composition of the stream flowing to the right with respect to one of the components is given by,

$$x_d = \frac{W'_h \cdot x_h - w'_h \cdot y_h}{W'_h - w'_h} \qquad \ldots.(17.38)$$

If the direction of flow of this component is towards the right, $x_d$ is positive, but if its direction of flow is to the left, $x_d$ is negative. For a countercurrent washing system, as shown in the diagram, the net flow of solvent at any stage will be to the left so that $x_{dS}$ is negative; the solute and insoluble residue will flow to the right making $x_{dA}$ and $x_{dB}$ positive.

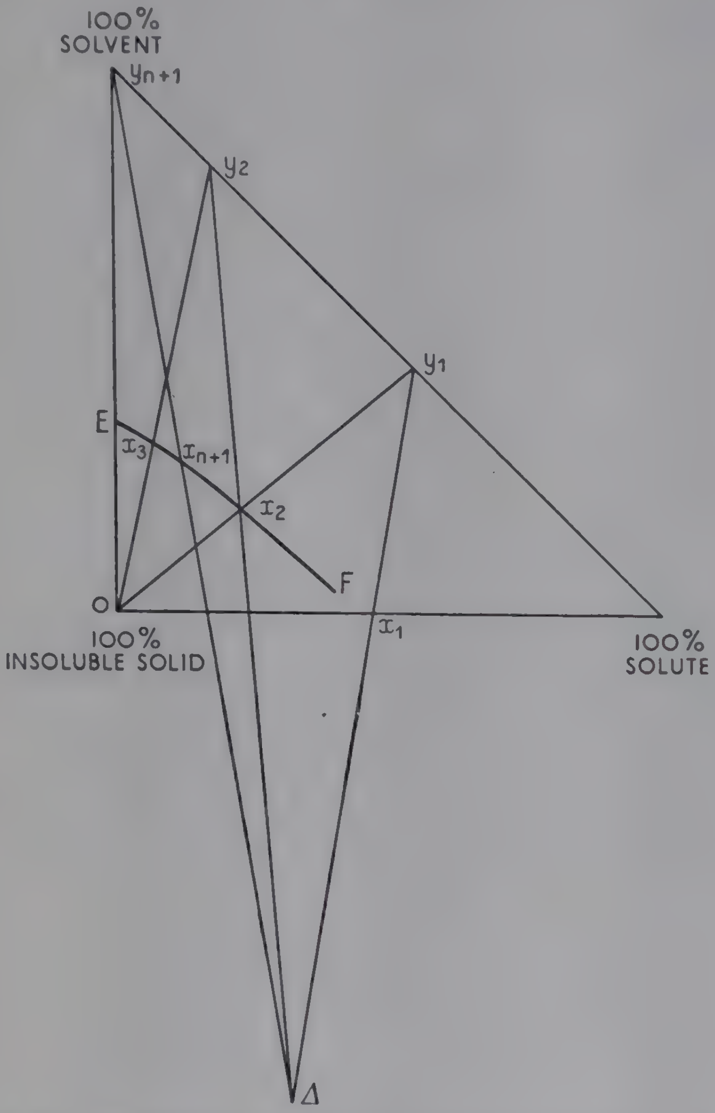

Fig. 17.23. Graphical method of solution with triangular diagram

Consider a system as shown in Fig. 17.22, in which a dry solid is extracted with a pure solvent. The compositions of the solid and solvent, and their flow rates, are specified. It is desired to wash the residual solid so that it has not less than a certain degree of purity, and to calculate the number of thickeners required to achieve this. The compositions of the solid to be extracted, the washed solid and the solvent ($x_1$, $x_{n+1}$ and $y_{n+1}$ respectively) are therefore given. The composition of the concentrated solution leaving the system $y_1$ can then be calculated from a material balance over the whole plant.

The difference point, which represents the composition of the net stream of material flowing to the right at all stages, must lie on the straight line through points $x_1$ and $y_1$ produced (Fig. 17.23). Point $x_1$ has co-ordinates ($x_{1A}$, $x_{1S}$); $x_{1A}$ is the composition of the dry solid fed to the system, and $x_{1S}$ is zero because this solid has no solvent associated with it. Point $y_1$ represents the composition of the concentrated solution discharged from the plant, and will lie on the hypotenuse of the triangle. The difference point will lie on the line through points $x_{n+1}$ and $y_{n+1}$. Now $x_{n+1}$, the composition of the final product in the underflow, will lie on the line *EF* which represents the compositions of all possible underflows and which is constructed from the experimental data on the amount of solution discharged in the underflow for various concentrations in the thickeners. Because pure solvent is used for washing, $y_{n+1}$ will lie at the top vertex of the triangle. The difference point is then obtained as the point of intersection of these two straight lines. It will be denoted by $\Delta$.

As the difference point represents the difference between the underflow and the overflow at any point in the system, it is now possible to calculate the compositions of all the streams, by considering each thickener in turn.

For the agitator and thickener 1, the underflow, of composition $x_2$, will contain insoluble solid mixed with solution of the same concentration as that in the overflow $y_1$, on the assumption that equilibrium conditions are reached in the thickener. All such mixtures of solution and insoluble solid are represented by compositions on the line $Oy_1$. As this stream is an underflow, its composition must also be given by a point on the line *EF*. Thus $x_2$ is given by the point of intersection of *EF* and $Oy_1$. The composition $y_2$ of the overflow stream from thickener 2 must lie on the hypotenuse of the triangle and also on the line through points $\Delta$ and $x_2$. The composition $y_2$ is therefore determined. In this manner it is possible to find the compositions of all the streams in the system. The procedure is repeated until the amount of solute in the underflow has been reduced to a value not greater than $x_{n+1}$, and the number of thickeners is then readily counted. For the simple example illustrated, only two thickeners would be required.

This method of calculation can be applied to any system, provided that streams of material do not enter or leave at some intermediate point. If the washing system alone were considered (such as that shown in Fig. 11.14), the insoluble solid would be introduced, not as fresh solid free of solvent, but as the underflow from the thickener in which the mixture from the agitator is separated. Thus $x_1$ would lie on the line *EF* instead of on the *A* axis of the diagram.

**Example.** Seeds, containing 20% by weight of oil, are extracted in a countercurrent plant, and 90% of the oil is recovered in a solution containing 50% by weight of oil. If the seeds are extracted with fresh solvent and 1 pound of solution is removed in the underflow in association with every two pounds of insoluble matter, how many ideal stages are required?

**Solution.** This problem will be solved using the graphical method.

Since the seeds contain 20% of oil,

$$x_{A1} = 0{\cdot}2 \quad \text{and} \quad x_{B1} = 0{\cdot}8$$

The final solution contains 50% of oil.

Thus

$$y_{A1} = 0{\cdot}5 \quad \text{and} \quad y_{S1} = 0{\cdot}5$$

The solvent which is used for extraction is pure and therefore

$$y_{S\,.\,n+1} = 1$$

Now each pound of insoluble solid in the washed product is associated with ½ pound of solution and 0·025 pounds of oil. Thus

$$x_{A\,.\,n+1} = 0{\cdot}0167, \qquad x_{B\,.\,n+1} = 0{\cdot}6667 \quad \text{and} \quad x_{S\,.\,n+1} = 0{\cdot}3166$$

The mass fraction of insoluble material in the underflow is constant and equal to 0·667. The composition of the underflow is therefore represented, on the diagram, by a straight line parallel to the hypotenuse of the triangle with an intercept of 0·333 on the two main axes.

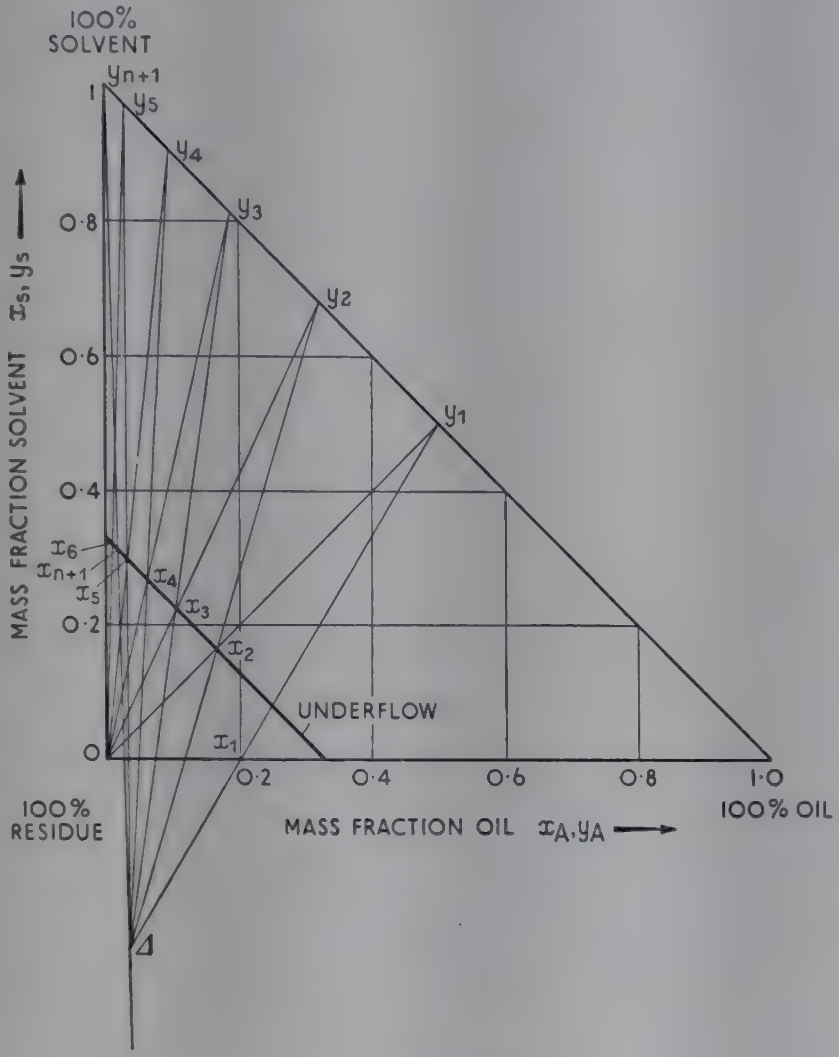
100%
SOLVENT
$y_{n+1}$
1
$y_5$
$y_4$
$y_3$
$y_2$
$y_1$
0·8
0·6
0·4
0·2
0
MASS FRACTION SOLVENT $x_S, y_S$
$x_6$
$x_{n+1}$
$x_5$
$x_4$
$x_3$
$x_2$
$x_1$
UNDERFLOW
0·2
0·4
0·6
0·8
1·0
100% OIL
100%
RESIDUE
MASS FRACTION OIL $x_A, y_A$
$\Delta$

Fig. 17.24.

The difference point is now found by drawing in the two lines connecting $x_1$ and $y_1$ and $x_{n+1}$ and $y_{n+1}$.

The graphical construction described in the text is then used and it is seen from the diagram (Fig. 17.24) that $x_{n+1}$ lies in between $x_5$ and $x_6$.

Thus five thickeners are adequate and produce the required degree of extraction.

## Non-Ideal Stages

If each stage in the extraction or washing system is not perfectly efficient, the ratio of solute to solvent in the overflow will be less than that in the underflow, and rather more units will be required than the number calculated by the method given. If the efficiency is independent of the concentration, allowance is simply made by dividing the theoretical number of ideal units by the efficiency. On the other hand, if the efficiency varies appreciably, this must be taken account of at each stage in the graphical construction. In Fig. 17.25, $x_h$ represents the composition of the underflow fed to thickener $h$, and this composition would be changed to $\bar{x}_{h+1}$ say in an ideal stage. The proportion of solute in the underflow is then reduced by an amount represented by $AB$. If the efficiency is less than unity, the change in the proportion of solute will be represented by $BC$, where $\frac{BC}{AB}$ is equal to the efficiency at the concentration considered, and the actual composition of the underflow is given by $x_{h+1}$. By this method it is possible to make allowance for a variable efficiency at each stage.

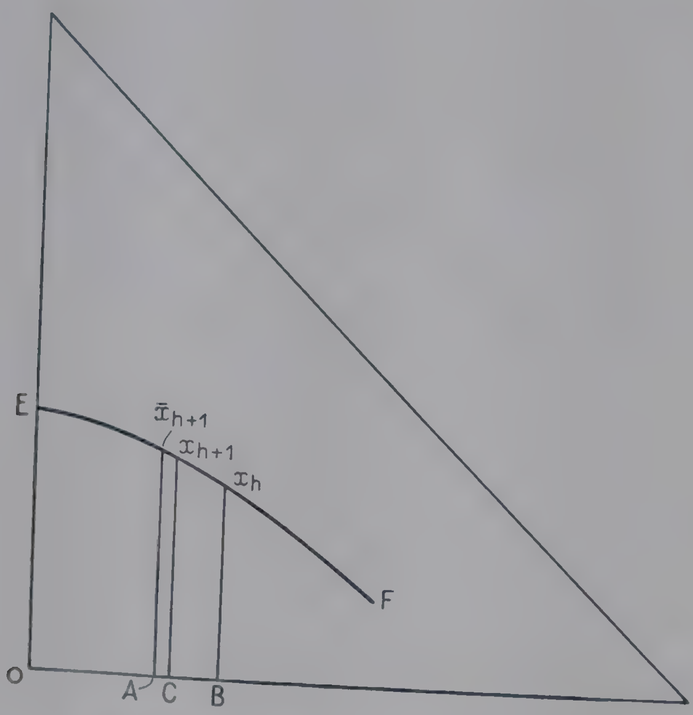

Fig. 17.25. Effect of stage efficiency

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## LIST OF SYMBOLS USED IN CHAPTER 17

| | | |
|---|---|---|
| $A$ | Area of solid-liquid interface | $\mathrm{L}^2$ |
| $a$ | Mass of solute per unit mass of solvent in final overflow | — |
| $b$ | Thickness of liquid film | L |
| $C_p$ | Specific heat of solution | — |
| $c$ | Concentration of solute in solvent | $\mathrm{ML}^{-3}$ |
| $c_0$ | Initial concentration of solute in solvent | $\mathrm{ML}^{-3}$ |
| $c_s$ | Concentration of solute in solvent in contact with solid | $\mathrm{ML}^{-3}$ |
| $D_L$ | Liquid phase diffusivity | $\mathrm{L}^2\mathrm{T}^{-1}$ |
| $F$ | Total mass of material fed to thickener | M |
| $F'$ | Difference between underflow and overflow | M |
| $f$ | Fraction of solute remaining with solids after washing | — |
| $h$ | Heat transfer coefficient | $\mathrm{ML}^{-2}\mathrm{T}^{-1}$ |
| $K$ | Solution per unit mass of insoluble solid in underflow | — |
| $K_L$ | Mass transfer coefficient | $\mathrm{LT}^{-1}$ |
| $k$ | Thermal conductivity | $\mathrm{ML}^{-1}\mathrm{T}^{-1}$ |
| $k'$ | A diffusion constant | $\mathrm{L}^2\mathrm{T}^{-1}$ |
| $L$ | Mass of solute in overflow per unit mass of insoluble | — |
| $M$ | Mass of solute transferred in time $t$ | M |
| $m$ | Number of batch washing thickeners | — |
| $N$ | Number of revolutions of stirrer in unit time | $\mathrm{T}^{-1}$ |
| $n$ | Number of countercurrent washing thickeners | — |
| $P$ | Mass of solute in overflow | M |
| $q$ | Insoluble in underflow per unit mass of solute in overflow | — |
| $R$ | Solvent ratio overflow: underflow | — |
| $R'$ | Ratio of solvent decanted to solvent retained | — |
| $S$ | Solute in underflow per unit mass insoluble | — |
| $s$ | Solvent in underflow per unit mass insoluble | — |
| $t$ | Time | T |
| $V$ | Volume of solvent used for extraction | $\mathrm{L}^3$ |
| $W$ | Mass of solution in underflow per unit mass insoluble | — |
| $W'$ | Total mass of underflow | M |
| $w$ | Mass of solution in overflow per unit mass of insoluble | — |
| $w'$ | Total mass of overflow | M |
| $X$ | Mass of solute per unit mass of solution | — |
| $x$ | Fractional composition of underflow | — |
| $x_d$ | Fractional composition of difference stream | — |
| $x'$ | Mass of solute per unit mass of solvent | — |
| $y$ | Fractional composition of overflow | — |
| $y''$ | Ratio of solute to solvent in overflow | — |
| $Z$ | Solvent in overflow per unit mass insoluble | — |
| $\mu$ | Viscosity of solution or liquid | $\mathrm{ML}^{-1}\mathrm{T}^{-1}$ |
| $\rho$ | Density of solution or liquid | $\mathrm{ML}^{-3}$ |

Suffixes

$A$, $B$, $S$ refer to solute, insoluble solid, solvent respectively.

1, . . . $h$, . . . $n$ refer to liquid overflow or underflow feed to units 1 . . . $h$, . . . $n$.

## CHAPTER 18

# *Distillation*

THE separation of mixtures of liquids into their component parts is one of the major processes of the chemical and petroleum industries, and distillation is the most widely used method of achieving this end. When a liquid containing two or more components is heated to its boiling point, the composition of the vapour will normally be different from that of the liquid. It is this difference in composition of the two phases at equilibrium that forms the basis of the distillation process, and for this reason a knowledge of the vapour-liquid equilibria is essential in the analytical treatment of a distillation problem. An important requirement of a distillation unit is the provision of intimate contact between the vapour and liquid streams so that equilibrium is approached. In practically no field of chemical engineering do the complete units vary so much in size, from the small laboratory units with a throughput of a few gallons per hour, to the giant stills of the petroleum industry handling several thousand gallons per hour.

## VAPOUR-LIQUID EQUILIBRIUM

The composition of the vapour in equilibrium with a liquid of given composition is determined experimentally using an equilibrium still. The results are conveniently shown on a temperature composition diagram (Fig. 18.1*a*, *b*, *c*). In the normal case (Fig. 18.1*a*), the curve *ABC* shows the composition of the liquid which boils at any given temperature, and the curve *ADC* the corresponding composition of the vapour at that temperature. Thus, a liquid of composition $x_1$ will boil at temperature $T_1$, and the vapour in equilibrium is indicated by point *D* of composition $y_1$. We can see that for any liquid composition $x$ the vapour formed will be richer in the more volatile component (where $x$ is the mol fraction of the more volatile component in the liquid, and $y$ in the vapour). Examples of mixtures giving this type of curve are benzene-toluene, *n*-heptane-toluene, and carbon disulphide-carbon tetrachloride.

In Fig. 18.1*b* and *c*, there is a critical liquid composition $x_g$ for which the vapour formed has the same composition, so that no change occurs on boiling. Such critical mixtures are called azeotropes, and special methods are necessary to effect separation. For compositions other than $x_g$, the vapour formed will have a different composition from that of the liquid. It is important to note that these diagrams are for constant pressure conditions, and that the composition of the vapour in equilibrium with a given liquid will change with pressure.

For distillation purposes it is more convenient to plot $y$ against $x$ at a constant pressure, since the majority of industrial distillations take place at substantially constant pressure. This is shown in Fig. 18.2 where it must be remembered that the temperature varies along each of the curves.

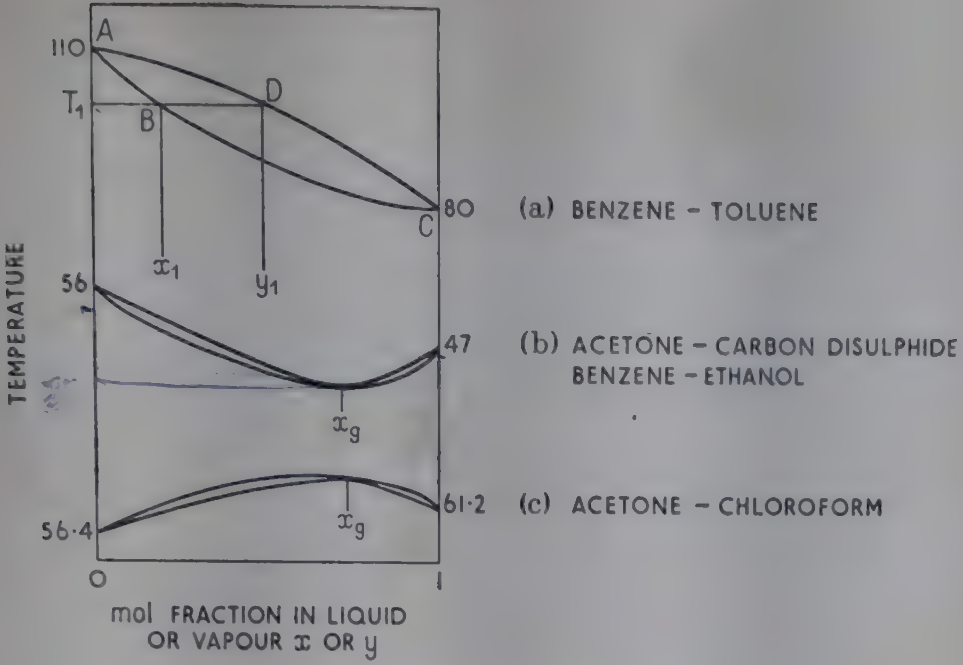


Fig. 18.1. Temperature-composition diagrams

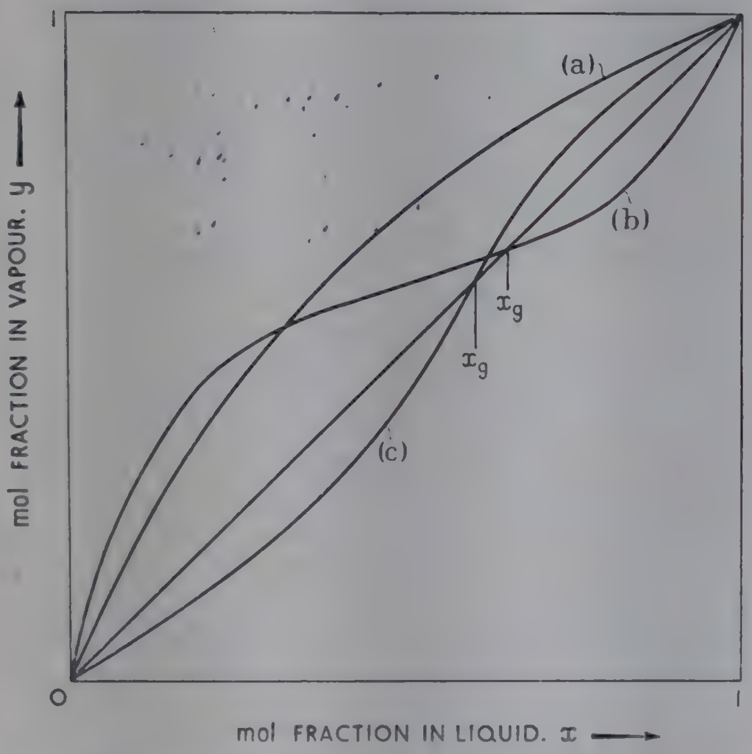


Fig. 18.2. Vapour composition as a function of liquid composition at constant pressure

## Partial Vaporisation and Partial Condensation

Suppose a mixture of benzene and toluene to be heated in a vessel, closed in such a way that the pressure remains atmospheric but no material can escape. If the mol fraction of the more volatile component (benzene) in the liquid is plotted as abscissa, and the temperature at which the mixture boils as ordinate, then the boiling curve is obtained as shown by *ABCJ* (Fig. 18.3). The corresponding dew point curve *ADEJ* shows the temperature at which a vapour of composition $y$ starts to condense.

If a mixture of composition $x_2$ is at a temperature below its boiling point ($T_3$), as shown by point *G* on the diagram, then on heating at constant pressure the following changes will occur:

1. When the temperature reaches $T_2$, the liquid will boil, as shown by point *B*, and some vapour of composition $y_2$, shown by point *E*, is formed.

2. On further heating the composition of the liquid will change, because of the loss of the more volatile component to the vapour. The boiling point will therefore rise to some temperature $T'$. At this temperature the liquid will have a composition represented by point *L*, and the vapour a composition represented by point *N*. Since no material is lost from the system, there will be a change in the proportion of liquid to vapour, the ratio being

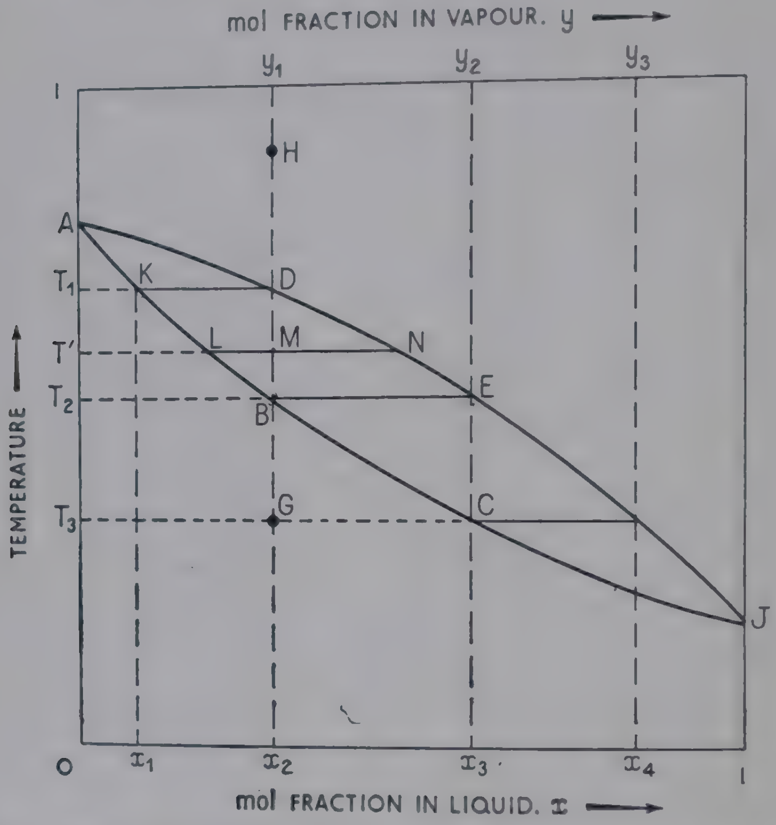


Fig. 18.3. Effect of partial vaporisation and condensation at the boiling point

$$\frac{\text{Liquid}}{\text{Vapour}} = \frac{MN}{ML}.$$

3. On further heating to a temperature $T_1$, all of the liquid is vaporised to give vapour *D* of the same composition $y_1$ as the original liquid.

We thus see that partial vaporisation of the liquid gives a vapour richer in the more volatile component than the liquid. If the vapour initially formed, as for instance at point *E*, is at once removed by condensation, then we obtain a liquid of composition $x_3$, represented by point *C*. The step *BEC* may be regarded as representing an ideal stage, since we pass from a liquid of composition $x_2$ to a liquid of composition $x_3$, which is a greater enrichment in the more volatile component than can be obtained by any other degree of vaporisation.

If we start with superheated vapour represented by point *H*, on cooling to *D* condensation will commence, and the first drop of liquid will have a composition *K*. Further cooling to $T'$ will give liquid *L* and vapour *N*. Thus, partial condensation brings about enrichment of the vapour in the more volatile component in the same manner as partial vaporisation. The industrial distillation column is in essence a series of units in which these two processes of partial vaporisation and partial condensation are effected simultaneously.

## Partial Pressures, Dalton's, Raoult's and Henry's Laws

The partial pressure $P_A$ of component *A* in a mixture of vapours is the pressure that would be exerted by component *A* at the same temperature, if present in the same volumetric concentration as in the mixture.

By Dalton's Law of Partial Pressures, $P = \Sigma P_A$, i.e. the total pressure is equal to the summation of the partial pressures. Then, in an ideal mixture, the partial pressure is proportional to the mol fraction of the constituent in the vapour phase,

i.e. $$P_A = y_A . P \qquad \ldots.(18.1)$$

A simple relation for the partial pressure developed by a liquid solute *A* in a solvent liquid *B* is given by Henry's Law in the form,

$$P_A = \mathscr{H}' . x_A \qquad \ldots.(18.2)$$

According to this law, the partial pressure is directly proportional to the mol fraction $x_A$ of the solute in the solvent. It is found that this holds only for dilute solutions.

The partial pressure is related to the concentration in the liquid phase by Raoult's Law which can be written

$$P_A = P_A{}^0 . x_A \qquad \ldots.(18.3)$$

where $P_A{}^0$ is the vapour pressure of pure *A* at the same temperature. This relation is usually found to be true only for high values of $x_A$, or correspondingly low values of $x_B$, but mixtures of organic isomers and some hydrocarbons follow the law closely. If *A* is regarded as the solute and *B* as the solvent, then Henry's Law applies when $x_A$ is small, and Raoult's Law when $x_B$ is small.

If the mixture follows Raoult's Law, then the vapour pressure of a mixture can be obtained graphically from a knowledge of the vapour pressure of the two components. Thus, in Fig. 18.4, *OA* represents the partial pressure $P_A$ of *A* in a mixture, and *CB* the partial pressure of *B*, the total pressure being shown by the line *BA*. Thus, in a mixture of composition *D*, the partial pressure $P_A$ is given by *DE*, $P_B$ by *DF*, and the total pressure *P* by *DG*.

Fig. 18.5 shows the partial pressure of one component *A* plotted against the mol fraction for a mixture that is not ideal. It will be found that over the range *OC* the mixture follows Henry's Law, and over *BA* it follows Raoult's Law. Although most mixtures show wide divergences from ideality, one of the laws is usually followed at very high and very low concentrations.

If it is known that the mixture follows Raoult's Law, then we can calculate the values of $y_A$ for various values of $x_A$ from a knowledge of the vapour pressures of the two components at various temperatures.

Thus,

$$P_A = P_A^\circ . x_A$$

and

$$P_A = P . y_A$$

so that

$$y_A = \frac{P_A^\circ . x_A}{P}, \quad \text{and } y_B = \frac{P_B^\circ . x_B}{P} \qquad \ldots .(18.4)$$

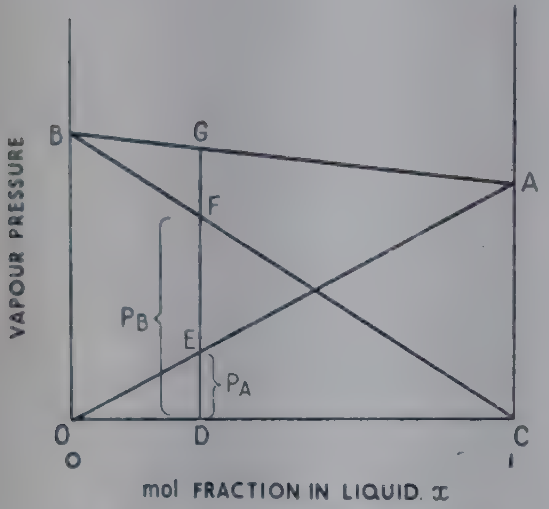


Fig. 18.4. Partial pressures of ideal mixtures

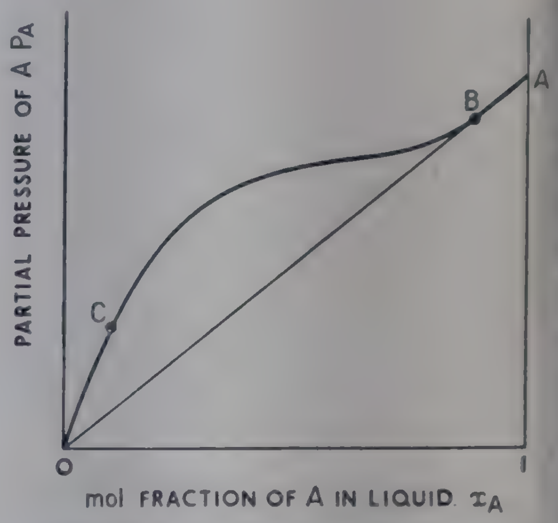


Fig. 18.5. Partial pressures of non-ideal mixtures

But

$$y_A + y_B = 1$$

$\therefore$

$$\frac{P_A^\circ . x_A}{P} + \frac{P_B^\circ . (1 - x_A)}{P} = 1,$$

giving

$$x_A = \frac{P - P_B^\circ}{P_A^\circ - P_B^\circ} \qquad \ldots .(18.5)$$

**Example.** For a mixture of *n*-heptane (*A*), and toluene (*B*), at 100°C and atmospheric pressure,

$$P_A^\circ = 795 \text{ mm of Hg},$$
$$P_B^\circ = 553 \text{ mm of Hg}.$$

$\therefore$

$$x_A = \frac{760 - 553}{795 - 553} = \underline{0{\cdot}856},$$

and

$$y_A = \frac{795 \times 0{\cdot}856}{760} = \underline{\underline{0{\cdot}896}}.$$

Equilibrium data usually have to be found by laborious laboratory methods. Some proposals have been made which enable the complete diagram to be deduced with reasonable accuracy from a relatively small number of experimental values. Some of these methods are discussed by ROBINSON and GILLILAND[5] and by THORNTON and GARNER[41].

## Relative Volatility ($\alpha$)

The relationship between the composition of the vapour $y_A$ and of the liquid $x_A$ in equilibrium can also be expressed in another way, which is particularly useful in distillation calculations. If we define the ratio of the partial pressure to the mol fraction in the liquid as the volatility, then we have

$$\text{volatility of } A = \frac{P_A}{x_A}, \text{ and}$$

$$\text{volatility of } B = \frac{P_B}{x_B}.$$

The ratio of these two volatilities is known as the relative volatility $\alpha$ given by

$$\boxed{\alpha = \frac{P_A \,.\, x_B}{x_A \,.\, P_B}}$$

Substituting $P \,.\, y_A$ for $P_A$, and $P \,.\, y_B$ for $P_B$, we get

$$\alpha = \frac{y_A \,.\, x_B}{y_B \,.\, x_A} \qquad \ldots .(18.6)$$

or

$$\frac{y_A}{y_B} = \alpha \,.\, \frac{x_A}{x_B} \qquad \ldots .(18.7)$$

This gives a valuable relation between the ratio of $A$ and $B$ in the vapour and the same ratio in the liquid.

Since with a binary mixture $y_B = 1 - y_A$, and $x_B = 1 - x_A$, we have

$$\alpha = \frac{y_A}{1 - y_A} \,.\, \frac{1 - x_A}{x_A}$$

or

$$y_A = \frac{\alpha \,.\, x_A}{1 + (\alpha - 1)x_A}. \qquad \ldots .(18.8)$$

This relation enables the composition of the vapour to be calculated for any desired value of $x$, if $\alpha$ is known.

From the definition of the volatility of a component, it is seen that for an ideal system the volatility is numerically equal to the vapour pressure of the pure component. Thus the relative volatility $\alpha$ may be expressed as

$$\alpha = \frac{P_A{}^0}{P_B{}^0} \qquad \ldots .(18.9)$$

This also follows by applying equation 18.1 from which $P_A/P_B = y_A/y_B$, so that

$$\alpha = \frac{P_A \,.\, x_B}{P_B \,.\, x_A} = \frac{P_A{}^0 \,.\, x_A \,.\, x_B}{P_B{}^0 \,.\, x_B \,.\, x_A} = \frac{P_A{}^0}{P_B{}^0}$$

Whilst $\alpha$ does vary somewhat with temperature, it remains remarkably steady for many systems, and a few values to illustrate this point are given below.

*For mixtures of $C_6H_6$ and $C_7H_8$*

| Temp. °C. | 80 | 90 | 100 | 110 |
|---|---|---|---|---|
| $\alpha$ | 2·62 | 2·44 | 2·40 | 2·39 |

## THE METHODS OF DISTILLATION—TWO COMPONENT MIXTURES

We can see from curve *a* of Fig. 18.2 that, for a binary mixture with a normal $y$–$x$ curve, the vapour is always richer in the more volatile component than the liquid from which it is formed. There are three main methods used in distillation practice which all rely on this basic fact;

1. Differential Distillation.
2. Flash or Equilibrium Distillation, and
3. Rectification.

Of these rectification is much the most important, and differs from the other two methods in that part of the vapour is condensed and returned as liquid to the still, whereas, in the other methods, all the vapour is either removed as such, or is condensed as product.

1. *Differential Distillation.* In this process the vapour formed on boiling the liquid is removed at once from the system. Since this vapour is richer in the more volatile component than the liquid, it follows that the liquid remaining becomes steadily weaker in this component, with the result that the composition of the product progressively alters. Thus, whilst the vapour formed over a short period is in equilibrium with the liquid, the total vapour formed is not in equilibrium with the residual liquid. The analysis of this distillation process was first put forward in 1902 by RAYLEIGH.

Let $S$ be the mols of material in the still and $x$ the fraction of component $A$.

Suppose an amount $\mathrm{d}S$ to be vaporised, containing a fraction $y$ of $A$.

Then a material balance on component $A$ gives,

$$y\,.\,\mathrm{d}S = \mathrm{d}(S\,.\,x),$$

$$= S\,.\,\mathrm{d}x\,.+x\mathrm{d}S,$$

$$\therefore \qquad \int_{S_0}^{S}\frac{\mathrm{d}S}{S} = \int_{x_0}^{x}\frac{\mathrm{d}x}{y-x}$$

and

$$\ln\frac{S}{S_0} = \int_{x_0}^{x}\frac{\mathrm{d}x}{y-x} \qquad \ldots.(18.10)$$

The integral on the right hand side can be solved graphically, if the equilibrium relation between $y$ and $x$ is available. In some cases a direct integration is possible. Thus, if over the range concerned, the equilibrium relation is a straight line of the form $y = mx + c$,

$$\ln \frac{S}{S_0} = \frac{1}{m-1} \ln \frac{(m-1)x + c}{(m-1)x_0 + c}$$

or

$$\frac{S}{S_0} = \left(\frac{y-x}{y_0 - x_0}\right)^{\frac{1}{m-1}}$$

i.e.

$$\frac{y-x}{y_0 - x_0} = \left(\frac{S}{S_0}\right)^{m-1} \qquad \ldots.(18.11)$$

From this equation we can calculate the amount of liquid to distil off, in order to obtain a liquid of given concentration in the still, and from this the average composition of the distillate can be found by a mass balance.

2. *Flash or Equilibrium Distillation.* This method is frequently carried out as a continuous process. The feed is passed into the still, where part is vaporised, the vapour remaining in contact with the liquid. The mixture of vapour and liquid leave the still and are separated so that the vapour is in equilibrium with the liquid. This system is widely used in the pipe stills of the petroleum industry, where, for instance, a crude oil enters a pipe still at 170°C and about 115 lbs/sq in., and leaves at 245°C and a pressure of only 43 lbs/sq in., some 15% being vaporised. An analysis of the process can be given as below, for a binary mixture of $A$ and $B$.

Let $V''$ be the mols of vapour formed, and $y$ the mol fraction of $A$,
$S$ be the mols of liquid, and $x$ the mol fraction of $A$, and
$F''$ be the mols of feed of mol fraction $x_f$ of $A$.

Then a material balance on the more volatile component $A$ gives

$$F'' . x_f = V'' . y + S . x$$

But

$$F'' = V'' + S$$

$\therefore$

$$\frac{V''}{F''} = \frac{x_f - x}{y - x} \qquad \ldots.(18.12)$$

3. *Rectification.* In the two processes outlined above, the vapour leaving the still at any time is in equilibrium with the liquid remaining, and there will normally be only a small increase in concentration of the more volatile component. The essential merit of rectification is that it enables a vapour to be obtained that is substantially richer than the liquid left in the still. This is achieved by an arrangement known as a fractionating column which enables successive vaporization and condensation to be accomplished in one unit.

### The Fractionating Column

The development of the fractionating column can be seen by considering three vessels, arranged as in Fig. 18.6, so that the vapour from the first (1) bubbles into the liquid in the second vessel (2), and the vapour from 2 passes into vessel 3. Steam is condensed in a closed coil within vessel 1 and the vapour from vessel 3 is totally condensed in the condenser. Suppose that the vessels 1, 2 and 3 initially contain liquids (benzene-toluene) of compositions $x_1$, $x_2$, $x_3$ as shown in Fig. 18.3. Then the following stages occur:

1. Liquid $x_1$ is boiled to give vapour $y_1$.
2. Vapour $y_1$ enters vessel 2 and, since it is at a higher temperature than the boiling point of liquid $x_2$, part of the vapour condenses and boils the liquid $x_2$ to give vapour $y_2$.
3. Vapour $y_2$ similarly condenses in vessel 3 to give vapour $y_3$.

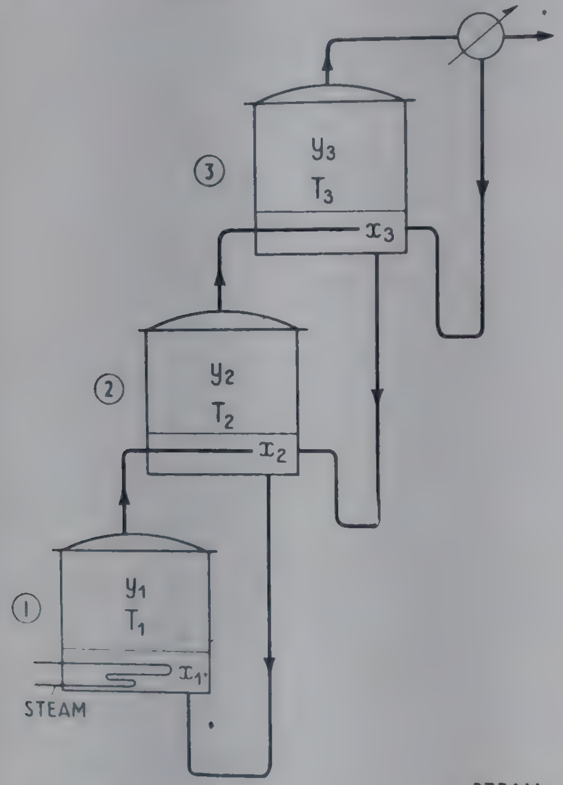


Fig. 18.6. A three-stage fractionation process

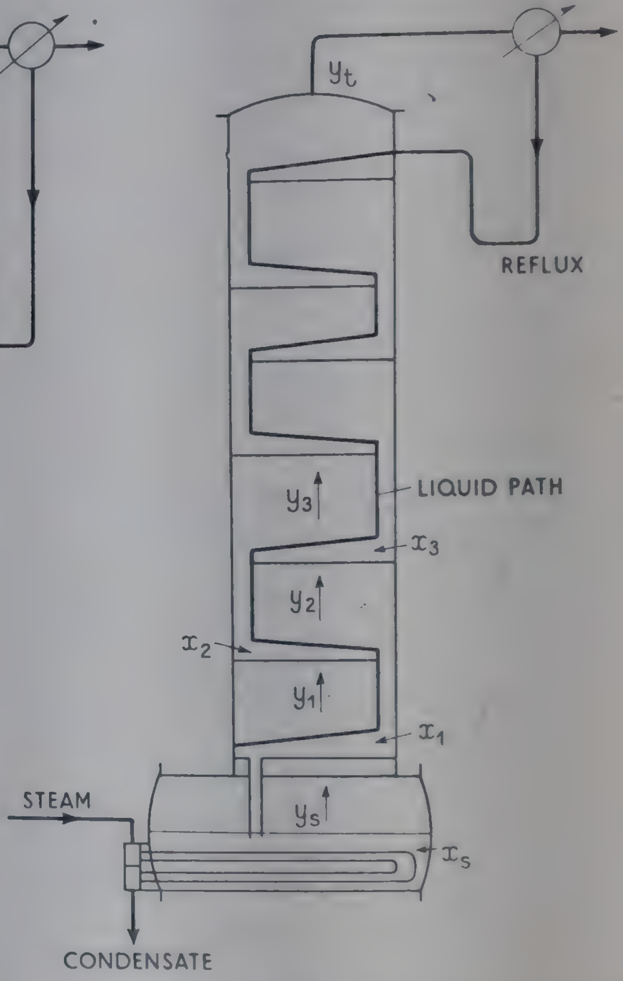


Fig. 18.7. A simple batch fractionating column

Since vapour $y_3$ is richer in benzene than the liquid $x_3$, the latter will be progressively weakened in benzene, and hence $y_3$ will steadily fall. If, however, we return some of the condensed product to vessel 3, then this will help to maintain the concentration of benzene in vessel 3. A material balance will show that, in order to keep the concentration up to the value of $x_3$, we shall have to return all of the product in the condenser and thus have no material leaving the still. In each vessel the vapour is partly condensed and a new vapour stream produced, giving conditions equivalent to continuous partial vaporization and condensation. Thus, each of the vessels can be considered as giving enrichment corresponding to an ideal step such as *BEC* in Fig. 18.3. It is important to notice that the heat required is supplied by the steam to vessel 1, and subsequently only by the rising vapour.

The conditions occurring in these vessels can be more neatly obtained by using a fractionating column, as shown in Fig. 18.7. Here a cylindrical vertical column is divided into a number of sections by a series of perforated trays which permit the upward flow of vapour. Liquid on each plate can run over a weir to the plate below. The column is connected at the top to a condenser, and at the bottom to a boiler which is usually heated by steam.

With this arrangement the condensing steam boils the liquid $x_s$ in the boiler giving vapour $y_s$ which enters the liquid $x_1$ on the first plate. Each of the sections thus forms a stage equivalent to one of the vessels in Fig. 18.6.

It is important to realise that the mass transfer between the vapour bubbles and the liquid on any tray occurs because these streams are not in equilibrium. Thus vapour ($y_1$) is passed into liquid ($x_2$), whereas vapour in equilibrium with $x_2$ would have composition $y_2$. On each plate the system tends to reach equilibrium because,

(a) Some of the less volatile component condenses from the rising vapour into the liquid, thus increasing the percentage of the more volatile component (M.V.C.) in the vapour.

(*b*) Some of the more volatile component is vaporised from the liquid on the plate thus decreasing the concentration of the M.V.C.

The number of molecules passing in each direction from vapour to liquid and in reverse will be approximately equal, since the heat given out by one molecule of the rising vapour on condensing is approximately equal to the heat required to vaporise one molecule from the liquid. The process of fractionation in an ideal case involves equimolecular counterdiffusion. In practice, equilibrium will not be attained between the vapour rising from a plate and the liquid remaining on the plate, so that the enrichment from one plate to the next will be less than that given by an ideal stage, as represented by *BEC* in Fig. 18.3. Again, as soon as any product is withdrawn from the top of the column, the enrichment per plate will fall, because insufficient of the M.V.C. is then returned from each stage to the one below. Frequently all of the vapour is condensed, and the fraction returned to the column is known as the reflux; the ratio of the amount returned to that removed as product is known as the reflux ratio, denoted by *R*. Instead of allowing the rising vapour to bubble through a perforated plate, the vapour may pass up through a series of riser tubes into small domes or caps from which it escapes into the liquid through a series of slots. This is known as a bubble cap plate and is discussed in detail on page 627.

A column apparatus may be used for batch or continuous operation. In the former case the column is mounted on a large boiler or still which holds the charge, and the column is said to be a rectifying column. The function of the plates is to increase the concentration of the more volatile component in the vapour and hence in the product, whilst the liquid remaining in the still becomes increasingly richer in the less volatile component. If the column is used in a continuous unit, the feed is usually admitted to some intermediate point in the column, the portion above the feed point being called the rectifying section, and that below, the stripping section. The plates below the feed strip out the more volatile component, so that the liquid withdrawn from the bottom of the column has a minimum concentration of this component, and a better recovery is obtained. Such an arrangement is indicated diagrammatically in Fig. 18.8, where it is seen that the feed *F* gives rise to the top product *D* and the bottom product *W*. The boiler in this case will be quite small, as it has to hold sufficient heating coils to vaporise only part of the liquid running from the bottom plate.

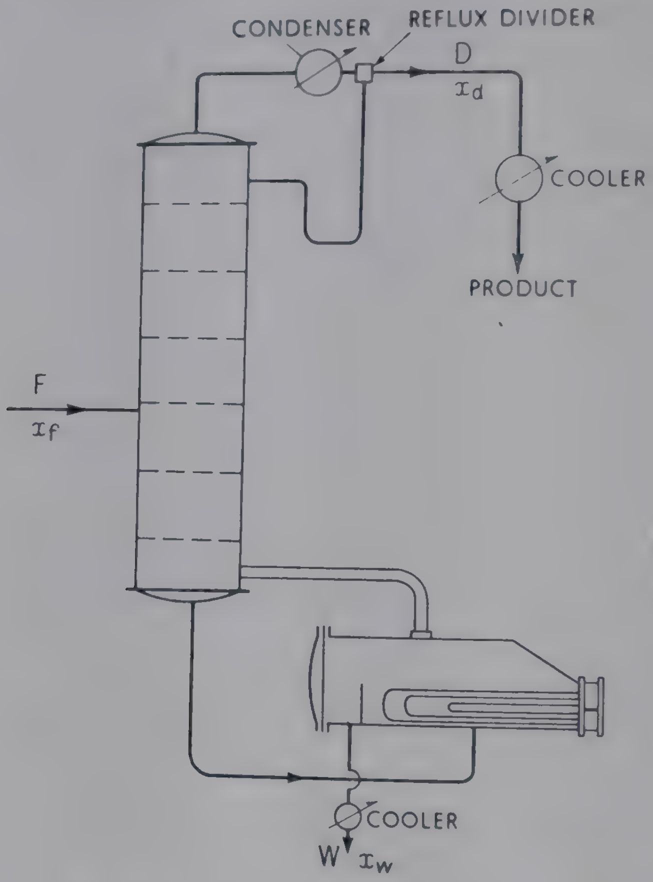


Fig. 18.8. A continuous fractionating column

A complete unit will normally consist of a feed tank, a feed heater, a column with boiler, a condenser, an arrangement for returning part of the condensed liquid as reflux, and coolers to cool the two products before running them to storage. The reflux liquor may be allowed to flow back by gravity to the top plate of the column or, as in the larger units of the petroleum industry, it is run back to a drum from which it is pumped to the top of the column. The control of the reflux on small units is conveniently done by hand operated valves, and with the larger units it is controlled by adjusting the delivery from a pump.

## Number of Plates Required in a Distillation Column

Four streams are involved in the transfer of heat and material across a plate, as shown in Fig. 18.9.

Plate $n$ receives liquid $L_{n+1}$ from plate above $(n + 1)$,
and vapour $V_{n-1}$ from plate below $(n - 1)$.
Plate $n$ supplies liquid $L_n$ to plate $n - 1$,
and vapour $V_n$ to plate $n + 1$.

The action of the plate is to bring about mixing so that the vapour $V_n$, of composition $y_n$, reaches equilibrium with the liquid $L_n$, of composition $x_n$. The streams $L_{n+1}$ and $V_{n-1}$ cannot be in equilibrium and, during the interchange process on the plate, some of the more volatile component is vaporized from the liquid $L_{n+1}$, decreasing its concentration to $x_n$, and some of the less volatile component is condensed from $V_{n-1}$, increasing the vapour concentration to $y_n$.

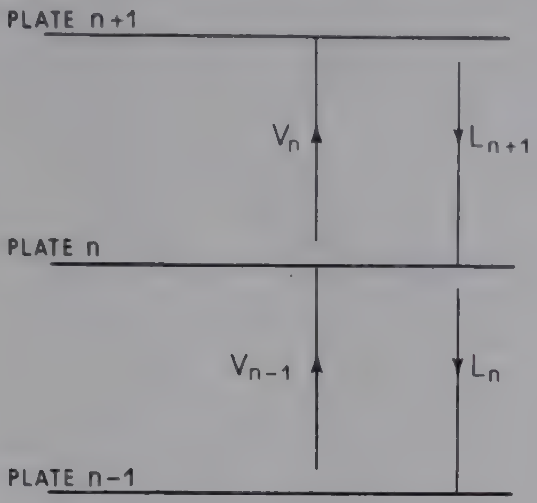


Fig. 18.9. Material balance over a plate

The heat to vaporize the more volatile component from the liquid is supplied by partial condensation of the vapour $V_{n-1}$. Thus the resulting effect is that the more volatile component is passed from the liquid running down the column to the vapour rising up, whilst the less volatile component is transferred in the opposite direction.

***Heat Balance over a Plate.*** If we take the datum temperature as that of the liquid on the plate, the only heat supplied to a plate $n$ is that from the rising vapour from plate $n - 1$. A heat balance across the plate can be written:—

$$L_{n+1} \cdot H^L_{n+1} + V_{n-1} \cdot H^V_{n-1} = V_n \cdot H^V_n + L_n \cdot H^L_n + \text{losses} + \text{heat of mixing} \quad \ldots.(18.13)$$

where $H^L_n$ is the enthalpy per mol of the liquid on plate $n$, and

$H^V_n$ is the enthalpy per mol of the vapour rising from plate $n$.

This equation is difficult to handle for the majority of mixtures, and some simplifying assumptions are usually made. Thus, with good lagging the radiation losses will be small and can be neglected, and for an ideal system the heat of mixing is zero. For such mixtures, Trouton's rule gives that the molal heat of vaporization divided by the temperature is constant, so that the molal heat of vaporization can be taken as constant and independent of the composition. Thus, one mole of vapour $V_{n-1}$ on condensing releases sufficient heat to liberate one mole of vapour $V_n$. It follows that $V_n = V_{n-1}$, so that the molal vapour flow is constant up the column unless material enters or is withdrawn from the section. The temperature change from one plate to the next will be small, and $H^L_n$ can be taken as equal to $H^L_{n+1}$. Applying these simplifications to equation 18.13, it is seen that $L_n$ equals $L_{n+1}$, so that the mols of liquid reflux are also constant in this section of the column. We thus have that $V_n$ and $L_n$ are constant over the rectifying section, and $V_m$ and $L_m$ over the stripping section.

For these conditions there are two methods for calculating the number of plates required to give a desired separation of a binary mixture, the first due to SOREL(1), and later modified by LEWIS(5a), and the second due to MCCABE and THIELE(8). The best account of the methods for calculating the required number of plates for distillation processes is given by UNDERWOOD(10).

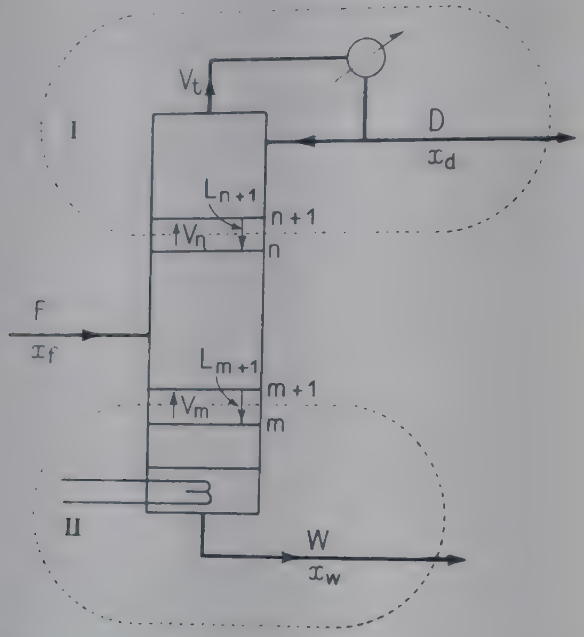


Fig. 18.10. Material balances at top and bottom of column

When the molal heat of vaporization varies appreciably, and the heat of mixing is no longer negligible, a different method of approach is necessary, as discussed on p. 659.

***Calculation of number of Plates using the Lewis-Sorel method.*** Suppose a unit is operating as shown in Fig. 18.10, so that a binary feed $F$ is distilled to give a top product $D$ and a bottom product $W$, with $x_f$, $x_d$, and $x_w$ as the corresponding mol fractions of the more volatile component. The vapour $V_t$ rising from the top plate is condensed, and part is run back as liquid at its boiling point to the column as reflux, the remainder being withdrawn as product.

A material balance above plate $n$ (indicated by the loop I in Fig. 18.10) gives

$$V_n = L_{n+1} + D \quad \text{....(18.14)}$$

Expressing this balance on the more volatile component, we have

$$y_n \,.\, V_n = L_{n+1} \,.\, x_{n+1} + D \,.\, x_d$$

Thus,

$$y_n = \frac{L_{n+1} \,.\, x_{n+1}}{V_n} + \frac{D \,.\, x_d}{V_n} \quad \text{....(18.15)}$$

This equation gives a relation between the composition of the vapour rising to the plate and the composition of the liquid on any plate above the feed plate. Since the mols of liquid overflow are constant, we can replace $L_{n+1}$ by $L_n$ and write

$$y_n = \frac{L_n \, . \, x_{n+1}}{V_n} + \frac{D \, . \, x_d}{V_n} \qquad \ldots.(18.16)$$

Similarly, taking a material balance for the total streams and for the more volatile component from the bottom to above plate $m$ (indicated by the loop II in Fig. 18.10), and noting that $L_m = L_{m+1}$ we have

$$L_m = V_m + W$$

and

$$y_m \, . \, V_m = L_m \, . \, x_{m+1} - W \, . \, x_w$$

$\therefore$

$$y_m = \frac{L_m \, . \, x_{m+1}}{V_m} - \frac{W \, . \, x_w}{V_m} \qquad \ldots.(18.17)$$

This is similar to equation 18.15, and gives the corresponding relation between the compositions of the vapour rising to a plate and the liquid on the plate, for the section below the feed plate. These two equations are the equations of the operating lines.

In order to calculate the change in composition from one plate to the next, we use the equilibrium data to find the composition of the vapour above the liquid, and the enrichment line to calculate the composition of the liquid on the next plate. This method can then be repeated up the column, using equation 18.17 for sections below the feed point, and equation 18.15 for sections above the feed point.

**Example.** A mixture of benzene and toluene containing 40 mol % of benzene is to be separated to give a product of 90 mol % of benzene at the top, and a bottom product with not more than 10 mol % of benzene. The feed is heated so that it enters the column at its boiling point, and the vapour leaving the column is condensed but not cooled, and provides reflux and product. It is proposed to operate the unit with a reflux ratio of 3 mols/mol product. It is required to find the number of theoretical plates needed and the position of entry for the feed. The equilibrium diagram for operating at 1 atmosphere pressure is shown in Fig. 18.11.

**Solution.** Considering 100 mols of feed a total material balance gives

$$100 = D + W$$

A balance on the M.V.C. (benzene) gives

$$100 \times 0{\cdot}4 = 0{\cdot}9 \, . \, D + 0{\cdot}1W$$

Thus

$$40 = 0{\cdot}9(100 - W) + 0{\cdot}1W$$

whence

$$W = 62{\cdot}5, \text{ and } \quad D = 37{\cdot}5$$

Using the notation in Fig. 18.10

$$L_n = 3D = 112{\cdot}5$$

and

$$V_n = L_n + D = 150$$

Thus, the top operating line from equation 18.15 is

$$y_n = \frac{112 \cdot 5}{150} \cdot x_{n+1} + \frac{37 \cdot 5 \times 0 \cdot 9}{150}$$

or

$$y_n = 0 \cdot 75\, x_{n+1} + 0 \cdot 225 \qquad \ldots .(18.18)$$

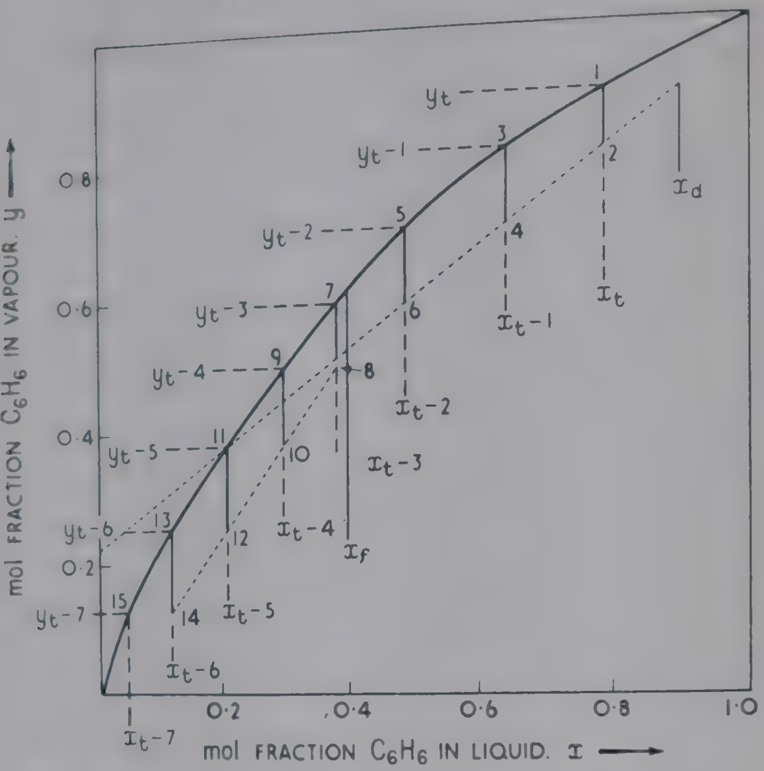


Fig. 18.11. Calculation of number of plates by the Sorel-Lewis method

Since the feed is all liquid at its boiling point, it will all run down as increased reflux to the plate below.

Thus,

$$L_m = L_n + F$$
$$= 112 \cdot 5 + 100 = 212 \cdot 5$$

Again,

$$V_m = L_m - W$$
$$= 212 \cdot 5 - 62 \cdot 5 = 150 = V_n$$

$$\therefore \qquad y_m = \frac{212 \cdot 5}{150} \cdot x_{m+1} - \frac{62 \cdot 5}{150} \times 0 \cdot 1$$

i.e.

$$y_m = 1 \cdot 415\, x_{m+1} - 0 \cdot 042 \qquad \ldots .(18.19)$$

With the two equations 18.18 and 18.19 and the equilibrium curve, the composition on the various plates can be calculated either by working from the still up to the condenser, or in the reverse direction. Since all the vapour from the column is condensed, the composition of the vapour $y_t$ from the top plate must equal that of the product $x_d$, and that of the liquor returned as reflux $x_r$. The composition $x_t$ of the liquor on the top plate is found from the equilibrium curve and since it is in equilibrium with vapour of composition $y_t = 0 \cdot 90$, $x_t = 0 \cdot 79$.

The value of $y_{t-1}$ is obtained from equation 18.18, as

$y_{t-1} = 0 \cdot 75 \times 0 \cdot 79 + 0 \cdot 225 = 0 \cdot 593 + 0 \cdot 225 = 0 \cdot 818$

$x_{t-1}$ is obtained from equilibrium curve as $0 \cdot 644$

$y_{t-2} = 0 \cdot 75 \times 0 \cdot 644 + 0 \cdot 225 = 0 \cdot 483 + 0 \cdot 225 = 0 \cdot 707$

$x_{t-2}$ from equilibrium curve $= 0 \cdot 492$

$y_{t-3} = 0 \cdot 75 \times 0 \cdot 492 + 0 \cdot 225 = 0 \cdot 369 + 0 \cdot 225 = 0 \cdot 594$

$x_{t-3}$ from equilibrium curve $= 0 \cdot 382$.

This is sufficiently near to that of the feed for the feed to be introduced on this plate. For the lower part of the column we shall use the operating line (18.19).

$\therefore$ $y_{t-4} = 1{\cdot}415 \times 0{\cdot}382 - 0{\cdot}042 = 0{\cdot}540 - 0{\cdot}042 = 0{\cdot}498$

$x_{t-4}$ from equilibrium curve $= 0{\cdot}298$

$y_{t-5} = 1{\cdot}415 \times 0{\cdot}298 - 0{\cdot}042 = 0{\cdot}421 - 0{\cdot}042 = 0{\cdot}379$

$x_{t-5}$ from equilibrium curve $= 0{\cdot}208$

$y_{t-6} = 1{\cdot}415 \times 0{\cdot}208 - 0{\cdot}042 = 0{\cdot}294 - 0{\cdot}042 = 0{\cdot}252$

$x_{t-6}$ from equilibrium curve $= 0{\cdot}120$

$y_{t-7} = 1{\cdot}415 \times 0{\cdot}120 - 0{\cdot}042 = 0{\cdot}169 - 0{\cdot}042 = 0{\cdot}127$

$x_{t-7}$ from equilibrium curve $= 0{\cdot}048$.

This liquor $x_{t-7}$ is slightly weaker than the minimum required and may be withdrawn as the bottom product. Thus, $x_{t-7}$ will correspond to the reboiler, and there will be seven plates in the column.

***The Method of McCabe and Thiele***(8). The simplifying assumptions of constant molal heat of vaporization, of no heat losses, and of no heat on mixing, led to a constant molal vapour flow and a constant molal reflux flow in any section of the column, i.e. $V_n = V_{n+1}$, $L_n = L_{n+1}$, etc. Using these simplifications we obtained the two enrichment equations, 18.16 and 18.17, as,

$$y_n = \frac{L_n}{V_n} \cdot x_{n+1} + \frac{D}{V_n} \cdot x_d, \qquad \ldots.(18.16)$$

and

$$y_m = \frac{L_m}{V_m} \cdot x_{m+1} - \frac{W}{V_m} \cdot x_w. \qquad \ldots.(18.17)$$

These equations were used in the Lewis-Sorel method to calculate the relation between the composition of the liquid on a plate and the composition of the vapour rising to that plate. McCabe and Thiele pointed out that, since these equations represent straight lines connecting $y_n$ with $x_{n+1}$ and $y_m$ with $x_{m+1}$, they can be drawn on the same diagram as the equilibrium curve to give a simple graphical solution for the number of stages required. Thus, the line of equation 18.16 will pass through the points 2, 4, 6, etc. shown in Fig. 18.11, and similarly the line of equation 18.17 will pass through points 8, 10, 12, 14.

In equation 18.16, if we put $x_{n+1} = x_d$, we get

$$y_n = \frac{L_n \cdot x_d}{V_n} + \frac{D \cdot x_d}{V_n} = x_d, \qquad \ldots.(18.20)$$

so that this equation represents a line passing through the point $y_n = x_{n+1} = x_d$. Further, if $x_{n+1}$ is put equal to zero, then $y_n = \frac{D \cdot x_d}{V_n}$, giving a second easily determined point. The top operating line is therefore drawn through two points $A$, $B$ of co-ordinates $(x_d, x_d)$ and $\left(O, \frac{D \cdot x_d}{V_n}\right)$.

For the bottom operating line, equation 18.17, if we put $x_m = x_w$, then

$$y_m = \frac{L_m \cdot x_w}{V_m} - \frac{W \cdot x_w}{V_m}.$$

Since $V_m = L_m - W$, it follows that $y_m = x_w$. Thus the bottom operating line passes through the point $C(x_w, x_w)$, and has a slope $L_m/V_m$. When the two operating lines have been drawn in, the number of stages required is found by drawing steps between the operating line and the equilibrium curve starting from point $A$.

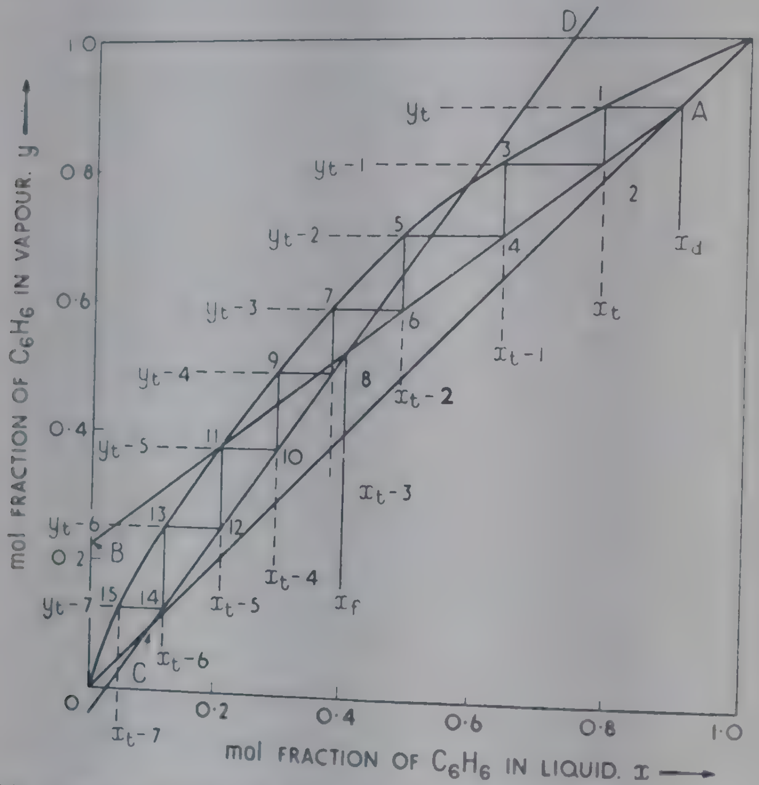


Fig. 18.12. Determination of number of plates by the McCabe-Thiele method

**Example—Using McCabe-Thiele method.** The previous example given on page 615 will now be worked using this method. Thus, with a feed composition $x_f$ of 0·4, the top composition $x_d$ is to have a value of 0·9 and the bottom composition $x_w$ of 0·10. The reflux $\frac{L_n}{D} = 3$.

**Solution** (Fig. 18.12).

1. By a material balance as given on p. 615 we get,

$$V_n = V_m = 150; \quad L_n = 112{\cdot}5; \quad L_m = 212{\cdot}5; \quad D = 37{\cdot}5; \quad W = 62{\cdot}5.$$

2. Draw the equilibrium curve and the diagonal line.
3. The equation of the top operating line is

$$y_n = 0{\cdot}75\, x_{n+1} + 0{\cdot}225 \qquad \ldots.(18.18)$$

Thus line $AB$ is drawn through the two points $A$ (0·9, 0·9) and $B$ (0, 0·225).

4. The equation of the bottom operating line is

$$y_m = 1{\cdot}415\, x_{m+1} - 0{\cdot}042 \qquad \ldots.(18.19)$$

This equation is represented by line $CD$ drawn through $C$ (0·1, 0·1) at a slope of 1·415.

5. Starting at point $A$, draw the horizontal line to cut the equilibrium curve on point 1. Drop the vertical through 1 to the operating line at point 2. Proceed in this way to obtain points 3–6.

6. Draw horizontal line through 6 to cut equilibrium curve on point 7, and vertical through 7 to lower enrichment line at point 8. Proceed to get points 9–16.

7. Count the number of stages, i.e., points 2, 4, 6, 8, 10, 12, 14, which gives the number of plates as 7.

***Enrichment in Still and Condenser.*** Point 16 in the example considered (Fig. 18.12) represents the concentration of the liquor in the still. The concentration of the vapour is represented by point 15, so that the enrichment represented by the increment 16–15 is achieved in the boiler or still body. Again, the concentration on the top plate is given by point 2, but the vapour from this plate has a concentration given by point 1, and the condenser by completely condensing the vapour gives a product of equal concentration, represented by point $A$. The still and condenser together, therefore, provide enrichment 16–15 + 1–$A$, which is equivalent to one ideal stage. Thus, the actual number of theoretical plates required is one less than the number of stages shown on the diagram. From a liquid in the still (point 16) to the product (point $A$) there are eight steps, but the column need only contain seven theoretical plates.

***The Intersection of the Operating Lines.*** It will have been seen from the example shown in Fig. 18.12 that the two operating lines intersect at a point having an $X$-co-ordinate of $x_f$. The locus of the point of intersection of the operating lines is of considerable importance since, as we shall see, it is dependent on the temperature and physical condition of the feed.

If the two operating lines intersect at a point with co-ordinates $(x_q, y_q)$ then we can write from equations 18.15, and 18.17

$$V_n \cdot y_q = L_n \cdot x_q + D \cdot x_d \qquad \ldots.(18.21)$$

and

$$V_m \cdot y_q = L_m \cdot x_q - W \cdot x_w \qquad \ldots.(18.22)$$

whence

$$y_q(V_m - V_n) = (L_m - L_n)x_q - (D \cdot x_d + W \cdot x_w) \qquad \ldots.(18.23)$$

A material balance over the feed plate gives

$$F + L_n + V_m = L_m + V_n$$

or

$$V_m - V_n = L_m - L_n - F. \qquad \ldots.(18.24)$$

To obtain a relation between $L_n$ and $L_m$, it is necessary to take an enthalpy balance over the feed plate, and to consider what happens when the feed enters the column. If the feed is all in the form of liquid at its boiling point, the reflux $L_m$ overflowing to the plate below will be $L_n + F$. If however the feed is a liquid at a temperature $T_f$, less than the boiling point, some vapour rising from the plate below will condense to provide sufficient heat to bring the feed liquor to the boiling point.

Let $H_f$ be the enthalpy per mol of feed, and $H_{fs}$ the enthalpy of one mol of feed at its boiling point. Then the heat to be supplied to bring feed to the boiling point is $F(H_{fs} - H_f)$, and the mols of vapour to be condensed to provide this heat is $\frac{F(H_{fs} - H_f)}{\lambda}$, where $\lambda$ is the molal latent heat of the vapour.

Then, reflux liquor

$$L_m = L_n + F + \frac{F(H_{fs} - H_f)}{\lambda}$$

$$= L_n + F\left(\frac{\lambda + H_{fs} - H_f}{\lambda}\right)$$

$$= L_n + q \, . \, F \qquad \ldots.(18.25)$$

where

$$q = \frac{\text{heat to vaporise 1 mol of feed}}{\text{molal latent heat of the feed}}$$

Thus from equation 18.24

$$V_m - V_n = q \, . \, F - F \qquad \ldots.(18.26)$$

A material balance of the more volatile component over the whole column gives

$$F \, . \, x_f = D \, . \, x_d + W \, . \, x_w.$$

Thus, from equation 18.23,

$$F(q - 1)y_q = qFx_q - Fx_f$$

or

$$y_q = \frac{q}{q-1} x_q - \frac{x_f}{q-1} \qquad \ldots.(18.27)$$

This equation is commonly known as the $q$-line, and shows that the point of intersection of the two operating lines lies on a straight line of slope $\frac{q}{q-1}$. If we put $x_q = x_f$, we see that $y_q = x_f$, and the $q$-line cuts the diagonal at $y = x_f$. Again, when $y_q = 0$, $x_q = x_f/q$, so that the line can be drawn through two points. From the definition of $q$, it follows that the slope of the $q$-line is governed by the nature of the feed.

| | | | |
|---|---|---|---|
| 1. Cold feed as liquor | $q > 1$ | $q$ line | / |
| 2. Feed at boiling point | $q = 1$ | $q$ line | \| |
| 3. Feed partly vapour | $0 < q < 1$ | $q$ line | \ |
| 4. Feed saturated vapour | $q = 0$ | $q$ line | — |
| 5. Feed superheated vapour | $q < 0$ | $q$ line | / |

These various conditions are indicated in Fig. 18.13.

Alteration in the slope of the $q$-line will alter the liquid concentration at which the two operating lines cut each other for a given reflux ratio. This will mean a slight alteration in the number of plates required for the given separation. Whilst the change in the number of plates will usually be rather small, if the feed is cold, there will be an increase in reflux flow below the feed plate, and hence an increased heat consumption from the boiler per mol of distillate.

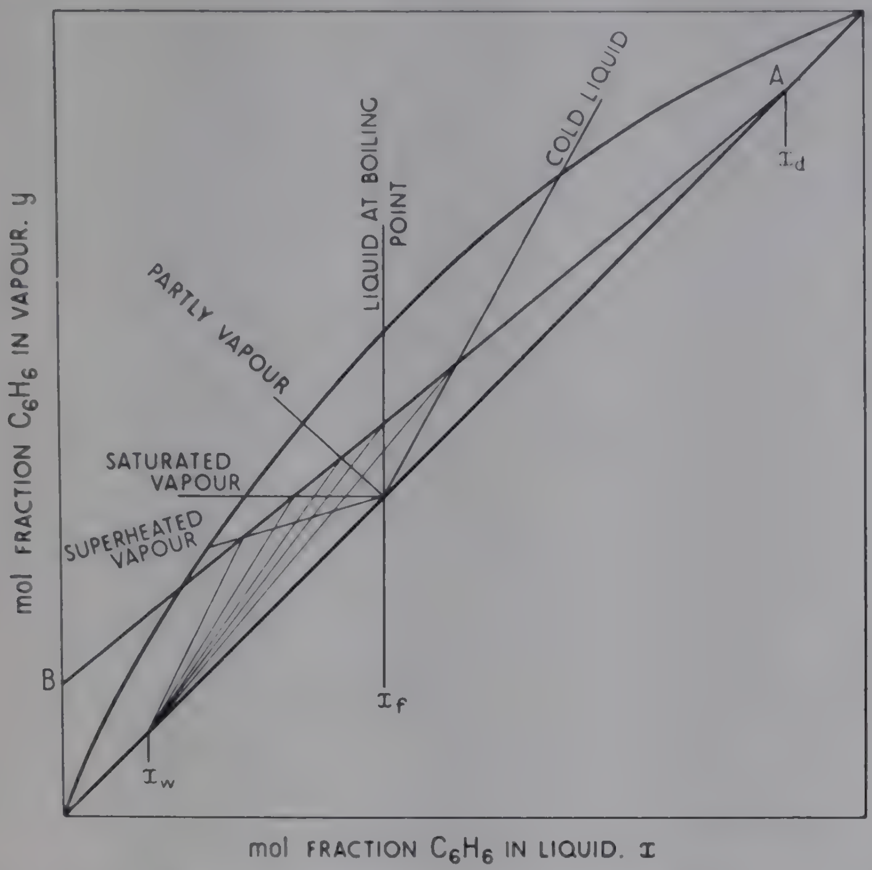

COLD LIQUID
A
$x_d$
LIQUID AT BOILING POINT
PARTLY VAPOUR
SATURATED VAPOUR
SUPERHEATED VAPOUR
B
$x_f$
$x_w$
mol FRACTION $C_6H_6$ IN VAPOUR. $y$
mol FRACTION $C_6H_6$ IN LIQUID. $x$

Fig. 18.13. Effect of the condition of the feed on the intersection of the operating lines for a fixed reflux ratio

## The Importance of the Reflux Ratio

***Influence on the Number of Plates required.*** The ratio $L_n/D$ of the top overflow to the quantity of product is denoted by $R$ and this enables the equation of the operating line to be expressed in another way, which is often more convenient. Thus, introducing $R$ in equation 18.16, we have:—

$$y_n = \frac{L_n}{L_n + D} \cdot x_{n+1} + \frac{D}{L_n + D} \cdot x_d \qquad \ldots.(18.28)$$

$$= \frac{R}{R+1} \cdot x_{n+1} + \frac{x_d}{R+1} \qquad \ldots.(18.29)$$

Any change in $R$ will therefore modify the slope of the operating line and, as can be seen from Fig. 18.12, will alter the number of plates required for a given separation. If $R$ is known, the top line is most easily drawn by joining point $A$ $(x_d, x_d)$ to $B\left(0, \frac{x_d}{R+1}\right)$ (Fig. 18.14). This method avoids the calculation of the actual flow rates $L_n, V_n$ when only the number of plates is to be estimated.

If no product is withdrawn from the still ($D = 0$), the column is said to operate under conditions of total reflux and, as seen from equation 18.28, the top operating line has its maximum slope of unity, and coincides with the line $x = y$. If the reflux ratio is reduced, the slope of the operating line is reduced and more stages are required to pass from $x_f$ to $x_d$, as shown by the line $AK$ in Fig. 18.14. Further reduction in $R$ will eventually bring the operating line to $AE$, when an infinite number of stages are needed to pass from $x_d$ to $x_f$. This arises from the fact that under these conditions the steps become very close together at liquid compositions near $x_f$, and no enrichment occurs from the feed plate to the plate above. These conditions are known as minimum reflux, and the reflux ratio is denoted by $R_m$. Any small increase in $R$ beyond $R_m$ will give a workable system, though a large number of plates will be required.

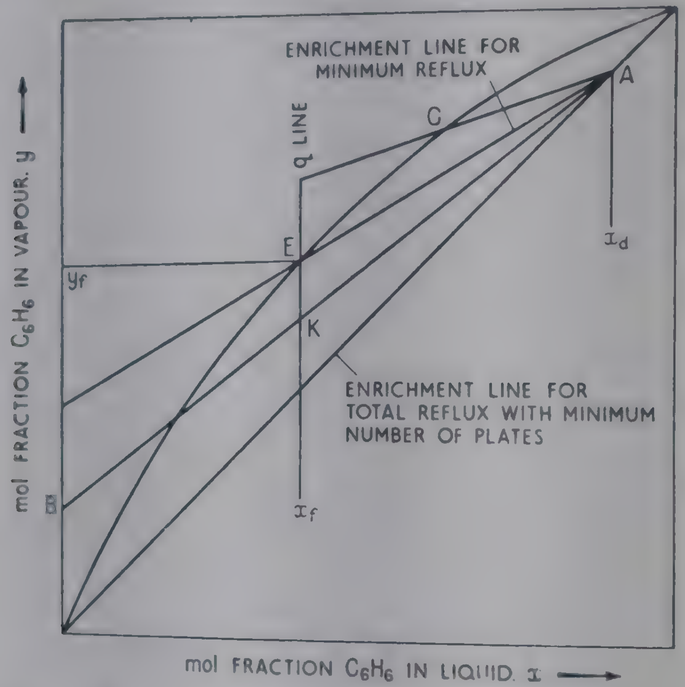

ENRICHMENT LINE FOR MINIMUM REFLUX
q LINE
G
A
E
$x_d$
$y_f$
K
ENRICHMENT LINE FOR TOTAL REFLUX WITH MINIMUM NUMBER OF PLATES
B
$x_f$
mol FRACTION $C_6H_6$ IN VAPOUR, y
mol FRACTION $C_6H_6$ IN LIQUID, x

Fig. 18.14. Influence of reflux ratio on the number of plates required for a given separation

It is important to note that any line such as $AG$, which is equivalent to a smaller value of $R$ than $R_m$, represents an impossible condition, since it is impossible to pass beyond point $G$ towards $x_f$. We thus arrive at two important deductions: first that the minimum number of plates is required for a given separation at conditions of total reflux, and secondly that there is a minimum reflux ratio below which it is impossible to obtain the desired enrichment, however many plates are used.

***Calculation of Minimum Reflux Ratio $R_m$.*** Fig. 18.14 represents conditions where the $q$-line is vertical, and the point $E$ lies on the equilibrium curve and has co-ordinates $(x_f, y_f)$. Then the slope of the line $AE$ is given by,

$$\frac{R_m}{R_m + 1} = \frac{x_d - y_f}{x_d - x_f}$$

or

$$R_m = \frac{x_d - y_f}{y_f - x_f} \qquad \ldots.(18.30)$$

If the *q*-line is horizontal as in Fig. 18.15, the enrichment line for minimum reflux is given by *AC*, where *C* has co-ordinates $(x_c, y_c)$.

Then

$$\frac{R_m}{R_m + 1} = \frac{x_d - y_c}{x_d - x_c}$$

or

$$R_m = \frac{x_d - y_c}{y_c - x_c} = \frac{x_d - x_f}{x_f - x_c} \qquad \ldots .(18.31)$$

since $y_c = x_f$.

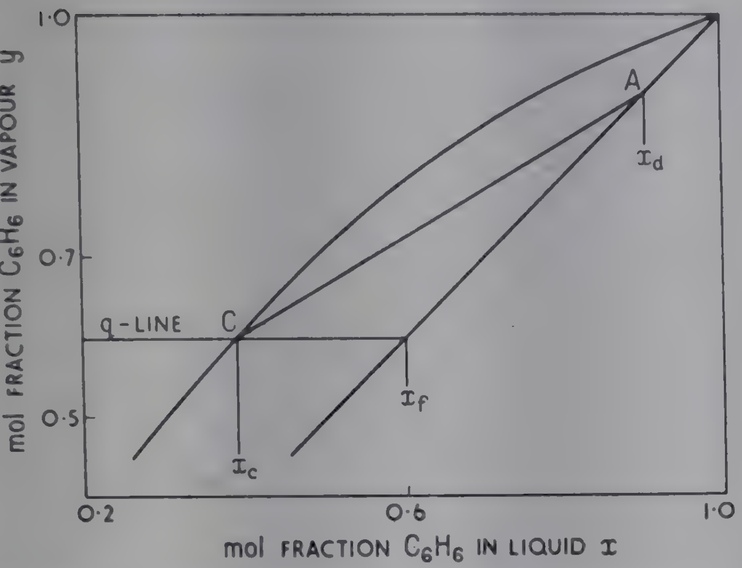


Fig. 18.15. Minimum reflux ratio with feed as saturated vapour

*Underwood and Fenske Equations.* For ideal mixtures, or where over the concentration range concerned the relative volatility can be taken as constant, $R_m$ can be obtained analytically from the physical properties of the system. Thus, if $x_{nA}$ and $x_{nB}$ are the mol fractions of two components *A* and *B* on any plate *n*, then a material balance over the top portion of the column above plate *n* gives,

$$V_n \,.\, y_{nA} = L_n \,.\, x_{(n+1)A} + D \,.\, x_{dA} \qquad \ldots .(18.32)$$

and

$$V_n \,.\, y_{nB} = L_n \,.\, x_{(n+1)B} + D \,.\, x_{dB} \qquad \ldots .(18.33)$$

Under conditions of minimum reflux, we have seen that a column has to have an infinite number of plates, or alternatively we can say that the composition on plate *n* is equal to that on plate *n* + 1. Dividing equation 18.32 by equation 18.33 and using the relations $x_{(n+1)A} = x_{nA}$ and $x_{(n+1)B} = x_{nB}$, we have

$$\frac{\alpha \,.\, x_{nA}}{x_{nB}} = \frac{y_{nA}}{y_{nB}} = \frac{L_n \,.\, x_{nA} + D \,.\, x_{dA}}{L_n \,.\, x_{nB} + D \,.\, x_{dB}}$$

$$\therefore \qquad R_m = \left(\frac{L_n}{D}\right)_{\min} = \frac{1}{\alpha - 1}\left[\frac{x_{dA}}{x_{nA}} - \alpha \,.\, \frac{x_{dB}}{x_{nB}}\right] \qquad \ldots .(18.34)$$

In this analysis, α is taken as the volatility of *A* relative to *B*. There is therefore, in general, a different value of $R_m$ for each plate. In order to produce any separation of the feed, the minimum relevant value of $R_m$ is that for the feed plate, so that the minimum reflux ratio for the desired separation is

$$R_m = \frac{1}{\alpha - 1}\left[\frac{x_{dA}}{x_{fA}} - \alpha \, . \, \frac{x_{dB}}{x_{fB}}\right] \qquad \ldots .(18.35)$$

for a binary system this becomes

$$R_m = \frac{1}{\alpha - 1}\left[\frac{x_{dA}}{x_{fA}} - \alpha \frac{(1 - x_{dA})}{(1 - x_{fA})}\right] \qquad \ldots .(18.36)$$

This relation could be obtained (from the geometrical relation), by putting $y = \frac{\alpha x}{1 + (\alpha - 1)x}$ (equation 18.8) in equation 18.30:

$$R_m = \frac{x_d - \dfrac{\alpha \, . \, x_f}{1 + (\alpha - 1)x_f}}{\dfrac{\alpha \, . \, x_f}{1 + (\alpha - 1)x_f} - x_f} = \frac{1}{\alpha - 1}\left[\frac{x_d}{x_f} - \frac{\alpha(1 - x_d)}{(1 - x_f)}\right] \qquad \ldots .(18.37)$$

***The Number of Plates at Total Reflux. Fenske's Method***(11). For conditions in which the relative volatility is constant, FENSKE derived an equation for calculating the required number of plates for a desired separation. Since no product is withdrawn from the still, the equations of the two operating lines become,

$$y_n = x_{n+1} \quad \text{and} \quad y_m = x_{m+1} \qquad \ldots .(18.38)$$

Consider two components $A$ and $B$, the concentrations of which in the still are $x_{sA}$ and $x_{sB}$. Then the composition on the first plate is obtained by,

$$\left(\frac{x_A}{x_B}\right)_1 = \left(\frac{y_A}{y_B}\right)_s = \alpha_s \left(\frac{x_A}{x_B}\right)_s$$

where the subscript outside the bracket indicates the plate, and $s$ the still. For plate 2,

$$\left(\frac{x_A}{x_B}\right)_2 = \left(\frac{y_A}{y_B}\right)_1 = \alpha_1 \left(\frac{x_A}{x_B}\right)_1 = \alpha_1 \, . \, \alpha_s \left(\frac{x_A}{x_B}\right)_s$$

And for plate $n$,

$$\left(\frac{x_A}{x_B}\right)_n = \left(\frac{y_A}{y_B}\right)_{n-1} = \alpha_1 \, . \, \alpha_2 \, . \, \alpha_3 \ldots \alpha_{n-1} \, . \, \alpha_s \, . \left(\frac{x_A}{x_B}\right)_s$$

If an average value of $\alpha$ is used, we get

$$\left(\frac{x_A}{x_B}\right)_n = \alpha_{av}^n \, . \left(\frac{x_A}{x_B}\right)_s$$

In most cases total condensation occurs in the condenser, so that

$$\left(\frac{x_A}{x_B}\right)_d = \left(\frac{y_A}{y_B}\right)_n = \alpha_n \cdot \left(\frac{x_A}{x_B}\right)_n = \alpha_{\mathrm{av}}^{n+1} \cdot \left(\frac{x_A}{x_B}\right)_s$$

$$\therefore \qquad n + 1 = \frac{\log \left(\frac{x_A}{x_B}\right)_d \cdot \left(\frac{x_B}{x_A}\right)_s}{\log \alpha_{\mathrm{av}}} \qquad \ldots.(18.39)$$

and $n$ is the required number of theoretical plates in the column.

It is important to note that, in this derivation, only the relative volatilities of two components have been used, and we shall see later (page 687) that the same relation can be applied to two components of a multicomponent mixture.

**Example.** For the separation of a mixture of benzene and toluene, as in the example on page 615, $x_d = 0{\cdot}9$, $x_w = 0{\cdot}1$, and $x_f = 0{\cdot}4$. Using an average value of 2·4 for the volatility of benzene relative to toluene, the number of plates required at total reflux is given by

$$n + 1 = \frac{\log \left(\frac{0{\cdot}9}{0{\cdot}1}\right)\left(\frac{0{\cdot}9}{0{\cdot}1}\right)}{\log 2{\cdot}4} = 5{\cdot}0$$

Thus the number of theoretical plates in the column is four, and this is independent of the composition of the feed.

If the feed is liquid at its boiling point, then the minimum reflux ratio $R_m$ is given by equation 18.37:—

$$R_m = \frac{1}{\alpha - 1}\left[\frac{x_d}{x_f} - \alpha\frac{(1 - x_d)}{(1 - x_f)}\right]$$

$$= \frac{1}{2{\cdot}4 - 1}\left[\frac{0{\cdot}9}{0{\cdot}4} - \frac{2{\cdot}4 \times 0{\cdot}1}{0{\cdot}6}\right]$$

$$= \underline{\underline{1{\cdot}32}}$$

Using the graphical construction shown in Fig. 18.15, since $y_f$ is 0·61, the value of $R_m$ is obtained as

$$R_m = \frac{x_d - y_f}{y_f - x_f} = \frac{0{\cdot}9 - 0{\cdot}61}{0{\cdot}61 - 0{\cdot}4} = \underline{\underline{1{\cdot}38}}$$

***Selection of Economic Reflux Ratio.*** The cost of a distillation stage may be considered as made up of the capital cost of the column, determined largely by the number and diameter of the plates, and the operating costs, determined by the steam and cooling water requirements. The depreciation charges can be taken as a percentage of the capital cost, and the two together taken as the overall charges. The steam required will be proportional to $V_m$, which can be taken as $V_n$ where the feed is liquid at its boiling point. From a material balance over the top portion of the column, $V_n = D(R + 1)$, so that the steam required per mol of product is proportional to $R + 1$; it will be a minimum when $R$ equals $R_m$, and will steadily rise as $R$ is increased.

The reduction in the required number of plates as $R$ is increased beyond $R_m$ will tend to reduce the cost of the column. For a column separating a benzene-toluene mixture, where $x_f = 0{\cdot}79$, $x_d = 0{\cdot}99$ and $x_w = 0{\cdot}01$ the number of theoretical plates as given by the McCabe-Thiele method are given below for various values of $R$. The minimum reflux ratio for this case is 0·81.

| Reflux Ratio $R$ | 0·9 | 1·0 | 1·1 | 1·2 |
|---|---|---|---|---|
| No. of Plates | 25 | 22 | 19 | 18 |

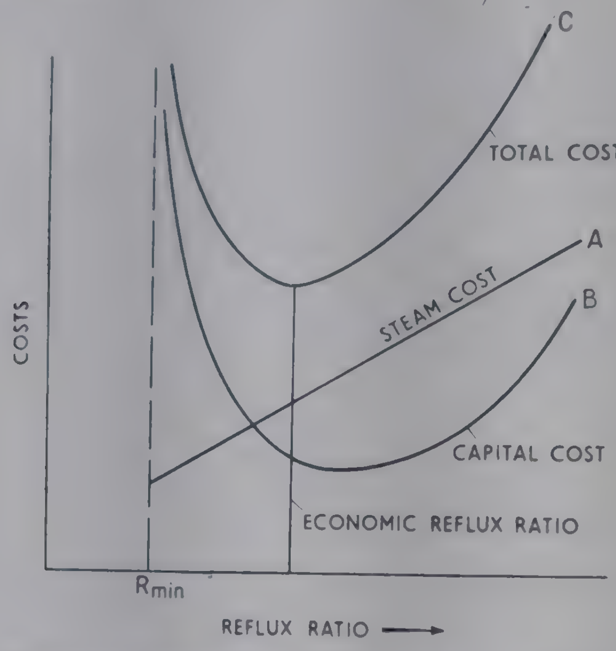


Fig. 18.16. Influence of reflux ratio on capital and operating costs of still

Thus, an increase in R, at values near $R_m$, gives a marked reduction in the number of plates, but at higher values of $R$, further increases have little effect on the number. Increasing the reflux ratio from $R_m$ therefore affects the capital and operating cost of a column in the following ways.

1. The operating costs rise and are approximately proportional to $R + 1$.
2. The capital cost initially falls since the number of plates falls off rapidly at this stage.
3. The capital cost rises at high values of $R$, since there is then only a very small reduction in the number of plates but the diameter, and hence area, continually increases because the vapour load becomes greater.

The total charges are found by adding the fixed and operating charges as shown in Fig. 18.16, where curve $A$ shows the steam costs and $B$ the fixed costs. The final picture is shown by curve $C$ which has a minimum value corresponding to the economic reflux ratio. There is no simple relation between $R_m$ and the optimum value, but practical values generally lie between 1·4 and 4 times the minimum.

### Location of Feed Point in a Continuous Still

From Fig. 18.17 we can see that, when stepping off plates down the top operating line $AB$, we cannot pass to the bottom operating line $CE$ until the value of $x_n$ on any plate is less than $x_e$. Again it is essential to pass to the lower line *CE* by the time $x_n = x_b$. The best conditions will be those where the minimum number of plates are used. From the geometry of the figure, we see that the largest steps in the enriching section occur down to the point of intersection of the operating lines at $x = x_q$. Below this value of $x$, the steps are larger on the lower operating line. Thus, although the column will operate for a feed composition between $x_e$ and $x_b$, the minimum number of plates will be required if $x_f = x_q$. For a binary mixture at its boiling point, this is equivalent to making $x_f$ equal the composition of the liquid on the feed plate.

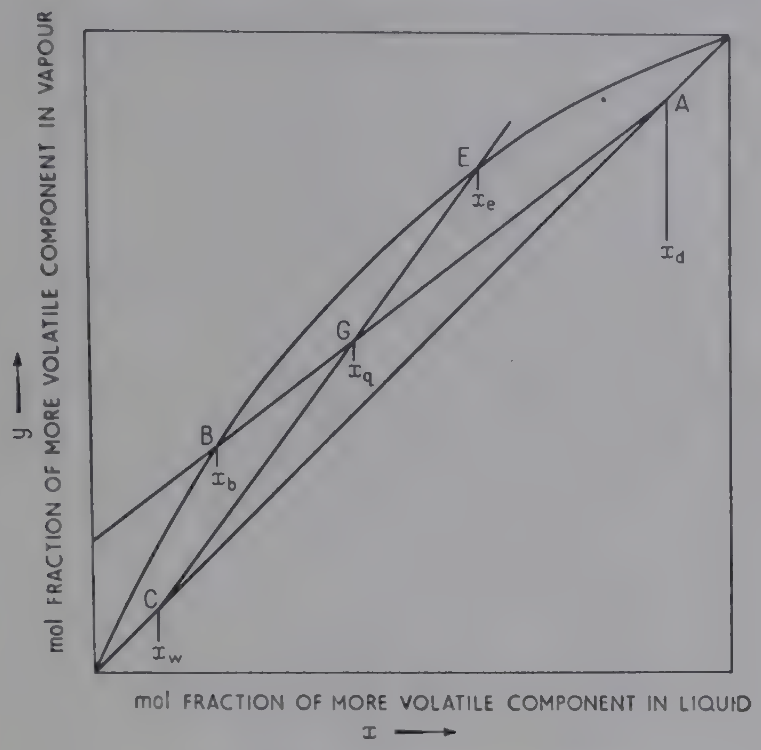

mol FRACTION OF MORE VOLATILE COMPONENT IN VAPOUR
y
A
E
$x_e$
$x_d$
G
$x_q$
B
$x_b$
C
$x_w$
mol FRACTION OF MORE VOLATILE COMPONENT IN LIQUID
x

Fig. 18.17. Location of feed point

## PLATE COLUMNS

The general construction of a bubble-cap plate is as illustrated in Fig. 18.18 The individual caps (Fig. 18.19) are mounted on risers and have either rectangular or triangular slots. They are held in place by some form of spider, the older method of using horizontal bars across a row leading to poor distribution of the liquor over the plate. The reflux passes to the plate below, either over two or three circular weirs and then down the attached pipes or, in large columns, over a straight weir and then down a segmental downcomer.

In considering the design of the column, the main requirements are high plate efficiency, the ability to handle the vapour and liquid streams, stability of operation, ease of erection and maintenance, and in many instances a small drop in pressure over each plate. Any tendency of the feed to deposit solids, or the possibility of charring, as with hydrocarbons, will influence the design. We shall first consider the meaning of plate efficiency and its derivation in terms of a mass transfer process, and then examine some of the main features which influence the mechanical layout of a plate and column.

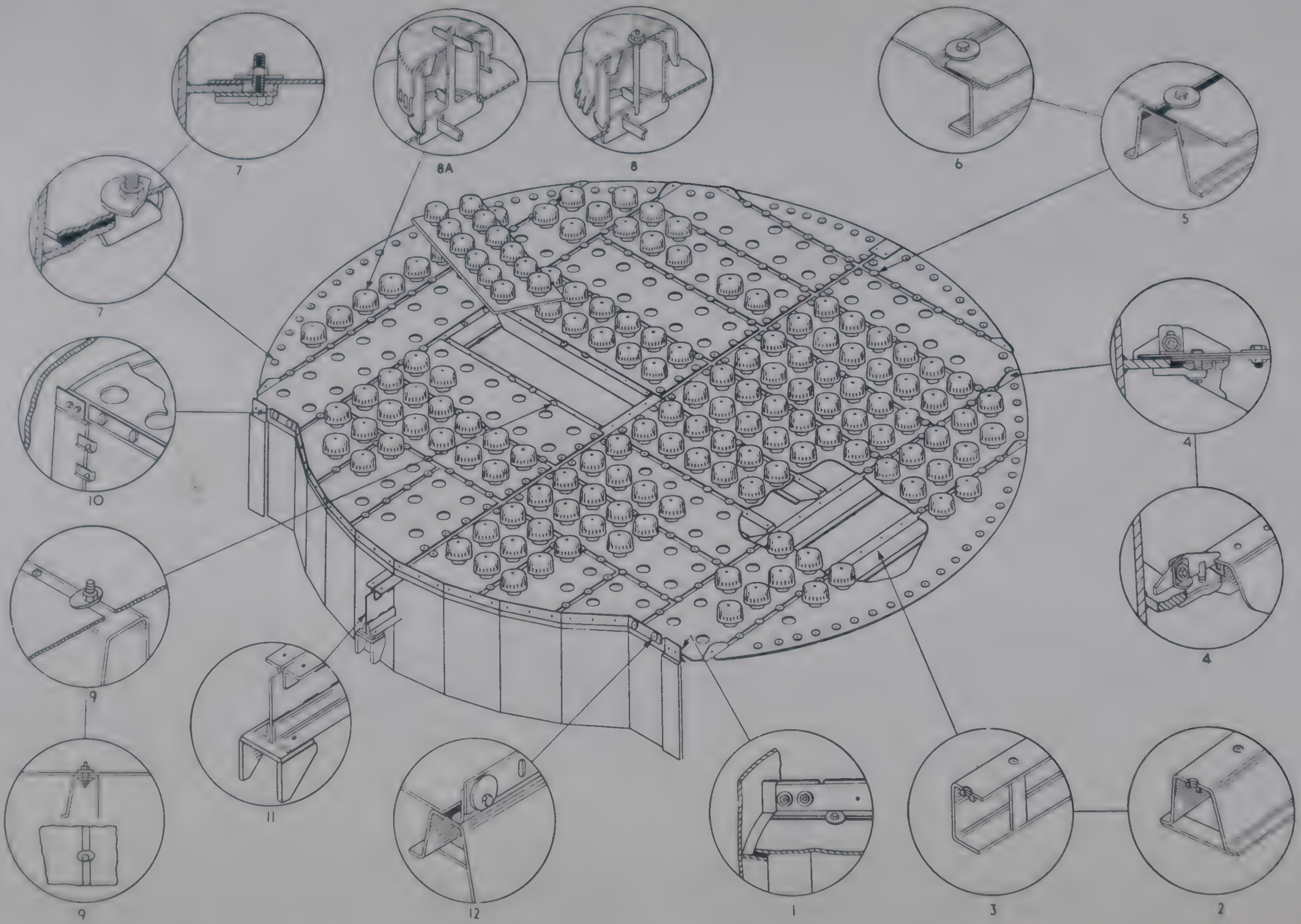

7
8A
8
6
5
7
10
4
4
9
11
9
12
1
3
2

Fig. 18.18. Construction of bubble-cap plate, showing engineering features of Glitsch "Truss-type" bubble trays

1 **Adjustable Weir Seal Plate**

Glitsch trays are provided with adjustable weir sea lplates at each end to ensure a liquid-tight seal between the end of the weir and the tower wall.

2 **Trapezoidal Minor Truss**

Designed to provide a flat surface on top with accessibility to the lower side, this shape allows maximum rigidity and strength at a minimum of material and labour cost. The neutral axis of the section is slightly above the centre line in order to reduce stresses at the top or compression side of the structure. Tension braces maintain the section modulus of the truss in the event of minor surges or explosions.

3 **Channel Minor Truss**

Like the trapezoidal truss, this type of minor truss is designed to afford a flat surface on top with easy accessibility to the lower side, with simplicity, economy and ruggedness as its important advantages. Attention is directed to the nuts which are welded to the underside of this type of truss as well as the trapezoidal truss.

4 **Top-Type Truss Hangers**

Built-up truss hangers are welded to the ends of the minor trusses to become integral parts of the trusses. The top-type hanger is provided with a clamping device for attaching the minor trusses to the tray support rings, thereby eliminating the necessity of individual brackets which otherwise would have to be welded to the tower wall. The truss hangers are also provided with individual clip angles to ensure that the outer edges of the tray floors do not tend to lift when load is applied to the tray.

5 **Thermal Expansion Joint between Tray Floors and Supporting Trusses**

Two important advantages are obtained from the use of frictional washers for holding down tray floor sections. Firstly, they provide for thermal expansion and contraction of the tray floor without risk of warping or buckling the relatively thin alloy sheet; the washers permitting independent lateral movement of all sections. Secondly, they eliminate the need for matching holes among component parts of the tray, with a consequent saving in fabrication and erection costs. The washers are bolted to nuts welded to the underside of the trusses (see diagrams 2 and 3).

6 **An Integral Minor Supporting Truss**

This method provides a minor truss section which is formed integrally with the tray floor.

7 **Peripheral Tray Clamp**

The clamps illustrated form a simple and effective means of holding the periphery of the tray to the tray support ring, and they may be installed from the upper side of the tray. Slotted holes in the outer tray floor sections compensate for "out-of-roundness" of the tower, and the clamps serve to prevent bending stress on the tray floors. The use of these clamps allows for expansion and contraction due to temperature. They also eliminate the need for drilling the tray support ring and for matching holes. The round frictional washer, plus if required an asbestos gasket, is provided as a means of sealing the slotted holes.

8 **Removable Cap and Riser Assembly**

This is one of the many unique Glitsch cap and riser assemblies. It incorporates a removable cap and riser, together with a "snap-in" frog hold-down device to permit speedy, simplified installation from the upper side of the tray. The cap is complete with internal riser lugs which add rigidity and ruggedness to the assembly. An extruded riser opening provides an excellent method of centring the riser and adds considerable rigidity to the tray floor.

8A The assembly is available either with a bolt and nut or with a wedge type hold-down, and both types are suitable for either round or rectangular riser openings.

9 **Hold-down Device for Manway Sections**

This Glitsch device provides accessibility from either side of the tray. The assembly consists of two manway lock clamps—one to be used on each side of the truss member, or the manway batten strip, in conjunction with a stud bolt. To free the manway section requires only loosening of the nut from either side and turning the entire assembly.

10 **Downcomer Clamping Bars**

Downcomer clamping bars are designed to effect a liquid and vapour tight seal at the outer edges of downcomers, and are usually welded to the tower wall by the tower manufacturers.

11 **Fabricated Major Beams**

On larger trays, usually greater than 10 ft diameter, where designs indicate heavy loads or explosion service, one or more major beams are provided. These beams are designed to meet the permissible maximum deflection allowances for loaded trays and to keep stresses within the allowable limit. Beams are attached to the tower wall by means of foot rests and vertical bolting plates, which can be welded to the tower wall either in the shop or in the field. When operating conditions permit, it is possible to hang these major supporting beams from the tower rings.

12 **Adjustable Weirs**

Provision for the alteration of weir levels is another useful feature frequently embodied in Glitsch trays in order to cater for possible process changes. In these cases weir plates are provided with slots to permit vertical adjustment.

**Plate Efficiency**

The efficiency of a plate is determined by a combination of the physical properties of the mixture and by the geometrical construction of the column. The efficiency is expressed either as an average for the column, or separately for each plate. Thus, if at a given reflux ratio a column with $n_p$ plates effects the same change in composition as would be achieved with $n$ theoretical plates, the average efficiency is $n/n_p$. The second method, due to MURPHREE[(9)], aims at expressing the efficiency of the individual plate and can be related to the mass transfer occurring on the plate.

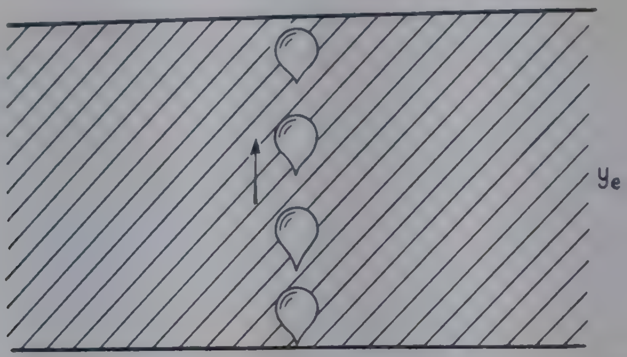


Fig. 18.20. Vapour bubbles passing through liquid

Suppose that the bubbles of vapour rising over a small area of the plate (Fig. 18.20) pass through the liquid in a time $t$, and that the average area of contact between the bubbles and the liquid is represented by $A'$. Then the rate equation for mass transfer for the more volatile component into the bubble can be written

$$K_g' \,.\, A' \,.\, (y_e - y)\, \mathrm{d}t = V' \,.\, \mathrm{d}y. \qquad \ldots .(18.40)$$

where,

$K_g'$ is the overall mass transfer coefficient,

$A'$ is the interfacial area offered by $V'$ mols of vapour,

$y, y_e$ are the mol fraction of a component in the gas phase and the mol fraction in the gas phase in equilibrium with the liquid phase, and

$V'$ denotes the mols of vapour as bubbles.

Integrating from $y_1$ the initial concentration to $y_2$ the exit concentration we get,

$$\int_{y_1}^{y_2} \frac{\mathrm{d}y}{y_e - y} = \int_0^t K_g' \,.\, \frac{A' \,.\, \mathrm{d}t}{V'}$$

Assuming $\frac{K_g' A'}{V'}$ to be independent of concentration, then as $y_e$ is constant on a given plate,

$$\ln \left[\frac{y_e - y_1}{y_e - y_2}\right] = \frac{K_g' \,.\, A' \,.\, t}{V'}$$

$$\therefore \qquad \ln \left[1 - \frac{y_2 - y_1}{y_e - y_1}\right] = -\frac{K_g' \,.\, A' \,.\, t}{V'}$$

$$\therefore \qquad \ln\,[1 - E_m] = -\frac{K_g' \,.\, A' \,.\, t}{V'} \qquad \ldots .(18.41)$$

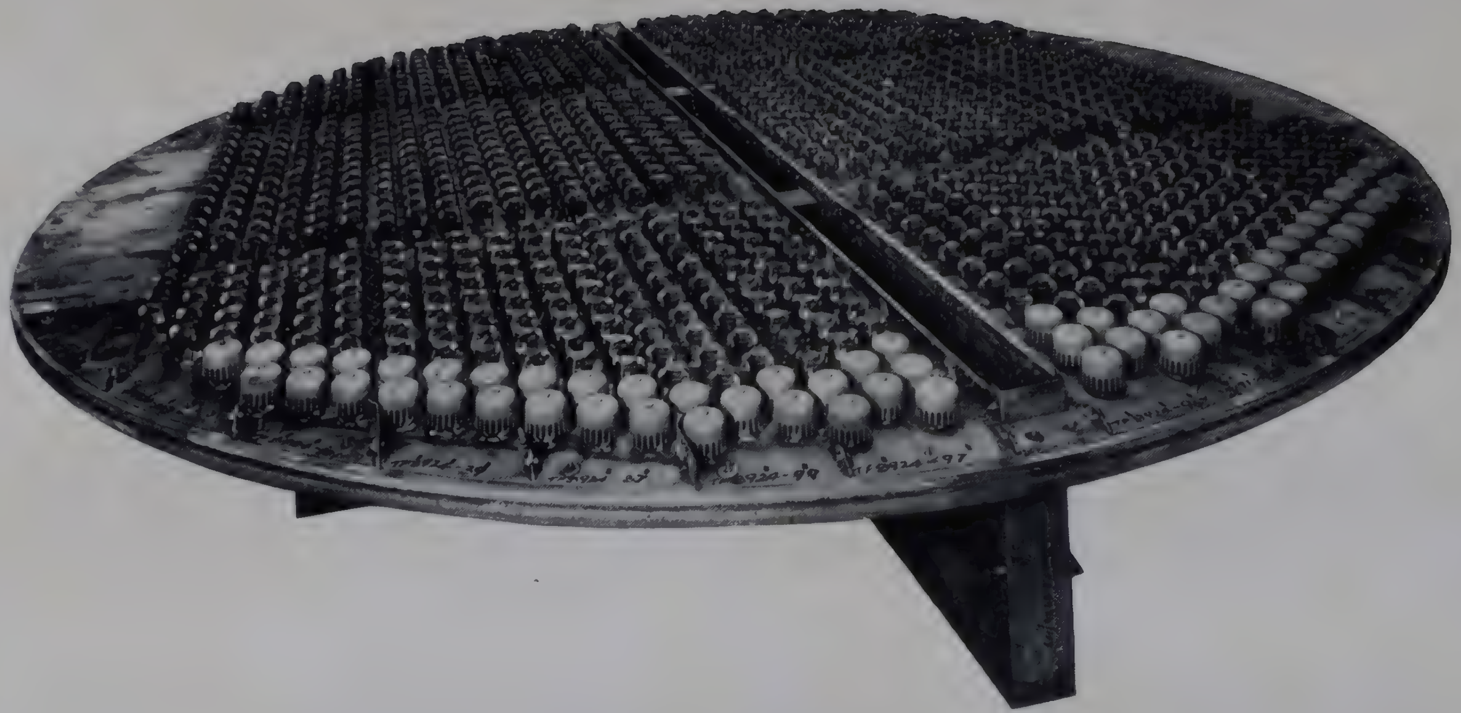


Fig. 18.19. 18 ft 6 in. diameter bubble tray (stainless steel) fitted with 1,118 4 in. diameter caps

Writing the transfer coefficient in terms of a driving force expressed as a molar concentration difference,

$$\ln[1 - E_m] = -\frac{K_g A' t}{V'} \cdot \frac{\rho_v}{M} \qquad \ldots.(18.41a)$$

where $\rho_v$ is the mean density of the vapour, and

$M$ is the mean molecular weight.

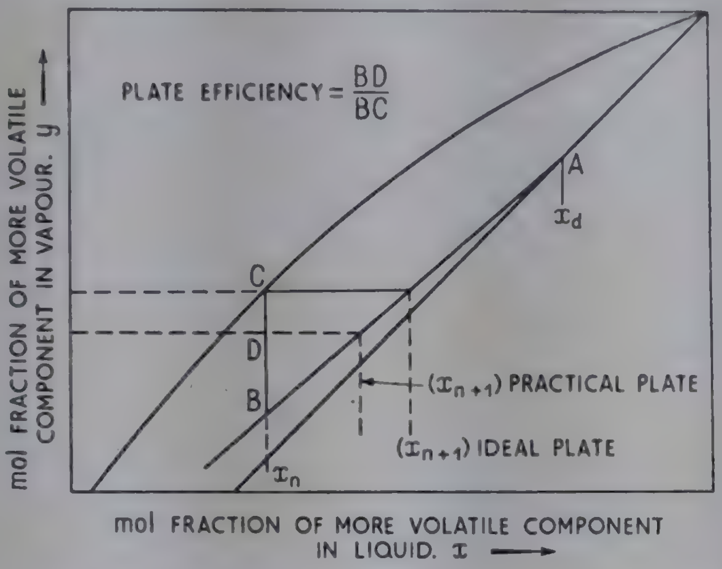


PLATE EFFICIENCY = BD/BC
A
$x_d$
C
D
B
$(x_{n+1})$ PRACTICAL PLATE
$(x_{n+1})$ IDEAL PLATE
$x_n$
mol FRACTION OF MORE VOLATILE COMPONENT IN VAPOUR. y
mol FRACTION OF MORE VOLATILE COMPONENT IN LIQUID. x

Fig. 18.21. Graphical representation of plate efficiency

From equation 18.41,

$$E_m = 1 - e^{-\frac{K_g' . A' . t}{V'}} \qquad \ldots.(18.42)$$

where $E_m = \dfrac{y_2 - y_1}{y_e - y_1}$ and is known as the local Murphree plate efficiency, and $t$ is the time of rise of the bubbles.

In this analysis the liquid surrounding the bubbles is assumed to have a constant composition. If average values for these concentrations are used, the group obtained represents the average Murphree plate efficiency $E$, which can be regarded as the ratio of the change in concentration achieved to that which would be obtained if equilibrium was attained.

$$E = \frac{y_{n+1} - y_n}{y_e - y_n} \qquad \ldots.(18.43)$$

where $y_n$, and $y_{n+1}$ are the average mol fractions of a component in the vapour leaving plates $n$ and $n + 1$.

This ratio for plate efficiency is shown graphically in Fig. 18.21. For the given operating line, $BC$ represents the enrichment that would be achieved with an ideal plate and $BD$ the actual enrichment, so that $BD/BC$ is the average plate efficiency.

While this treatment gives a useful indication of the efficiency of each plate, it involves a number of assumptions that are known to be only partly true. Thus $K_g$ is much higher at the formation of the bubble than during its rise through the liquid (Chapter 20), and the bubbles are neither spherical, nor distinct. There will be a concentration gradient in the liquid across the plate and this influences the rate of transfer. The composition of the liquid stream on mixing with the vapour from the first row of caps is changed so that the transfer between the liquid and the vapour from the second row of caps will be reduced. Thus, if the vapour leaves in equilibrium with the liquid at the centre of the plate, it will be richer than that corresponding to equilibrium with the liquid leaving the plate. Practical measurements of plate efficiency may, therefore, give values greater than 100%.

### *Expressions for Plate Efficiency related to Mass Transfer*

Several attempts have been made to derive an expression for the plate efficiency from a knowledge of the physical and geometrical properties of the system. GEDDES[33a] assumed that the bubbles were spherical and, putting $\frac{V'}{A'M\rho_v}$ equal to one sixth of the bubble diameter $d$, equation 18.41 becomes

$$\ln(1 - E_m) = \frac{-6\,.\,K_g\,.\,t}{d} \qquad \text{....(18.44)}$$

where $\rho_v$ is the density of the vapour and $M$ is the mean molecular weight.

The overall transfer coefficient $K_g$ can be expressed in terms of the film coefficients $k_g$ and $k_l$ by the relation,

$$\frac{1}{K_g} = \frac{1}{k_g} + \frac{m}{k_l} \qquad \text{....(18.45)}$$

where $m$ is the slope of the equilibrium curve;

i.e.

$$m = \frac{\mathrm{d}y_e}{\mathrm{d}x} \qquad \text{....(18.46)}$$

The local efficiency $E_m$ is then found from equation 18.44 by expressing $k_g$, $k_l$ and $d$ in terms of the physical properties of the system and the flow rates.

*Bubble Diameter d.* This can be found by relating the buoyancy force on the bubble leaving the orifice to the surface tension,

$$\frac{4}{3}\,.\,\pi\,.\left(\frac{d}{2}\right)^3.\,[\rho_L - \rho_v]\,g = 2\,.\,\pi\,.\,r\,.\,\sigma \qquad \text{....(18.47)}$$

where $r$ is the orifice radius,

$\sigma$ is the surface tension, and

$\rho_L, \rho_v$ are the densities of the liquid and vapour.

Thus,

$$\frac{d}{2} = 1{\cdot}14\left[\frac{r\,.\,\sigma}{g[\rho_L - \rho_v]}\right]^{1/3} \qquad \text{....(18.48)}$$

For a vertical slot with height $h_s$ and width $w$, equation 18.47 can be written for a low gas flow as

$$\frac{4}{3}\,.\,\pi\,.\left(\frac{d}{2}\right)^3.\,(\rho_L - \rho_v)g = 2\,.\,\sigma\,.\,[h_s + w] \qquad \text{....(18.49)}$$

or

$$\frac{d}{2} = 0{\cdot}78\left[\frac{(h_s + w)\sigma}{g[\rho_L - \rho_v]}\right]^{1/3} \qquad \text{....(18.50)}$$

For a more rapid flow of gas, the constant is taken as unity, so that

$$\frac{d}{2} = \left[\frac{(h_s + w)\sigma}{g[\rho_L - \rho_v]}\right]^{1/3} \qquad \text{....(18.51)}$$

*Transfer Coefficient* $k_y$. This is calculated on the assumption of unsteady state diffusion between the liquid and the vapour. $k_g$ can be obtained from relations developed by Barrer(3),

$$k_g = \frac{-\left(\frac{d}{2}\right)}{3 \,.\, t} \ln \left\{ \frac{6}{\pi^2} \sum_{N=1}^{N=\infty} \frac{1}{N^2} \,.\, e^{-\frac{D_V \,.\, N^2 \pi^2 \,.\, t}{(d/2^2)}} \right\} \qquad \ldots .(18.52)$$

where $D_V$ is the diffusivity in the vapour phase, and

$t$ is the average time of contact of the bubble and the liquid (submergence divided by bubble velocity).

*Transfer Coefficient* $k_l$. This has been taken from the work of Higbie(18), discussed in Chapter 20.

$$k_l = 2 \,.\, \left[\frac{D_L}{\pi \,.\, t_e}\right]^{1/2} \qquad \ldots .(18.53)$$

where $D_L$ is the diffusivity in the liquid phase, and

$t_e$ is the time of existence of the liquid surface taken as bubble diameter divided by velocity.

The values obtained from equations 18.51, 52, and 53 are then used in equations 18.44 and 18.45 to give the local plate efficiency $E_m$. Geddes showed examples of these calculations and obtained good agreement with experimental data.

Chu(42) has worked on the same general basis as Geddes, but has taken different equations to determine $k_g$, $k_l$, and $d$. Thus, on the basis of Colburn's analogy between heat and mass transfer, he evaluates the film coefficients from the equations

$$k_g = \frac{0{\cdot}023 \,.\, u_a}{\left[\frac{d \,.\, \rho_v \,.\, u_a}{\mu_v}\right]^{\cdot 2} \,.\, \left[\frac{\mu_v}{\rho_v \,.\, D_V}\right]^{2/3}} \qquad \ldots .(18.54)$$

where $\rho_v$ is the density of the vapour,
$d$ is the diameter of bubble,
$u_a$ is the ascending velocity of bubble,
$\mu_v$ is the viscosity of vapour,
$D_V$ is the gas diffusivity, and
$k_g$ is the gas film transfer coefficient,

and $k_l$ is obtained from a similar equation using $\rho_L$, $\mu_L$, $D_L$ as corresponding properties for the liquid phase.

For evaluating the equivalent diameter he takes the relationship given by van Krevelen(40):

$$d = 0{\cdot}024 \left[\frac{V^2 \,.\, T^2 \rho_L}{P^2[\rho_L - \rho_v]}\right]^{1/5} \qquad \ldots .(18.55)$$

where $d$ is the diameter of the bubble in ft,
$\rho_L$,$\rho_v$ are the densities of the liquid and vapour in lb/ft$^3$,
$V$ is the vapour flow in lb-mols/hr,
$T$ is the absolute temperature in °$R$,
$P$ is the pressure in atmospheres.

The velocity of rise of the bubble $u_a$ is taken as $\frac{(gd)^{\frac{1}{2}}}{2}$, and the time of contact of bubble and liquid from the total liquid depth and the velocity $u_a$.

The local efficiency $E_m$ is then obtained from equation 18.44, $K_g$ being obtained by substituting the values of $k_g$ and $k_l$ in equation 18.45.

Bakowski(44) assumed that the vapour film controlled the process and that the bubble flow was in the form of chain bubbles. The flow of the bubbles is then considered as equivalent to the flow in a wetted wall column and $k_g$ is taken as

$$k_g = \frac{b_1 \,.\, u^{0\cdot75}}{P^{0\cdot25} \,.\, d^{0\cdot25}} \qquad \ldots.(18.56)$$

where $u$ is the vapour velocity in cm/sec.,

$d$ is the equivalent diameter of the channel in cm,

$P$ is the pressure in atmospheres, and

$k_g$ is in g-mol/sec-cm$^2$-atm.

$b_1$ was assumed proportional to the difference between the molecular flow rates at the surface under the actual and equilibrium conditions. He presented a final expression for plate efficiency in the form

$$\log_{10} \frac{1}{1 - E_m} = \frac{0\cdot34 h_L T^{0\cdot5}}{M^{0\cdot5} \,.\, v_s{}^{0\cdot25} \,.\, P^{0\cdot25}} \qquad \ldots.(18.57)$$

It is assumed that the mean cross sectional area of the channel is constant and that the vapour velocity is proportional to the vapour rate per slot. The interfacial area is taken as proportional to the depth of submergence $h_L$ (taken here as cms). In equation 18.57, $v_s$ is the vapour rate per slot in cc/sec.,

$M$ is the molecular weight of the vapour entering plate,

$T$ is the temperature °K, and

$h_L$ is the depth of liquid above the bottom of the slot in cm.

These equations are rather too difficult to apply because of the lack of physical data but they do indicate the factors considered to influence the diffusional interchange between a vapour and a liquid. They do illustrate the difficulty in applying the apparently simple two-film theory to practical problems.

***Empirical Relations for Plate Efficiency.*** An empirical relation for the overall plate efficiency of fractionating columns operating with petroleum hydrocarbons has been given by Drickamer and Bradford(30). They correlated their results in the following form, in which the only physical property used is the average viscosity of the liquid feed.

$$E = 0\cdot17 - 0.616 \log_{10} \sum x_f \,.\, \mu_L \qquad \ldots.(18.58)$$

where $x_f$ is the mol fraction of the component in the feed, and

$\mu_L$ is its viscosity in centipoises at the mean tower temperature.

**Example.**

| Component | Mol Fraction in feed $x_f$ | $\mu_L$ at 190° $F$ | $\mu \,.\, x_f$ |
|---|---|---|---|
| $C_3$ | 0·2 | 0·048 | 0·0096 |
| $C_4$ | 0·3 | 0·112 | 0.0336 |
| $C_5$ | 0·2 | 0·145 | 0·0290 |
| $C_6$ | 0·3 | 0·188 | 0·0564 |
| | | | 0·1286 |

Then $E = 0\cdot17 - 0\cdot616 \log 0\cdot1286$
$= 0\cdot72.$

The towers studied varied in diameter from 4 to 7½ ft, with riser areas of 7·2 to 11·5% of the tower area, and the caps had slots ¾ in. to 1¼ in. deep and up to ¼ in. in width.

Later, O'CONNELL$^{(34a)}$ found that an empirical relationship could be given for a wider range of liquors by including the relative volatility of the key components (see page 675). His relation is shown in Fig. 18.22. CHU$^{(42)}$, in addition to presenting the complex analysis previously considered, gave the following simplified equation,

$$\log E = 1{\cdot}67 + 0{\cdot}30 \log (L/V) - 0{\cdot}25 \log (\mu_L \alpha) + 0{\cdot}092 h_L \quad \ldots .(18.59)$$

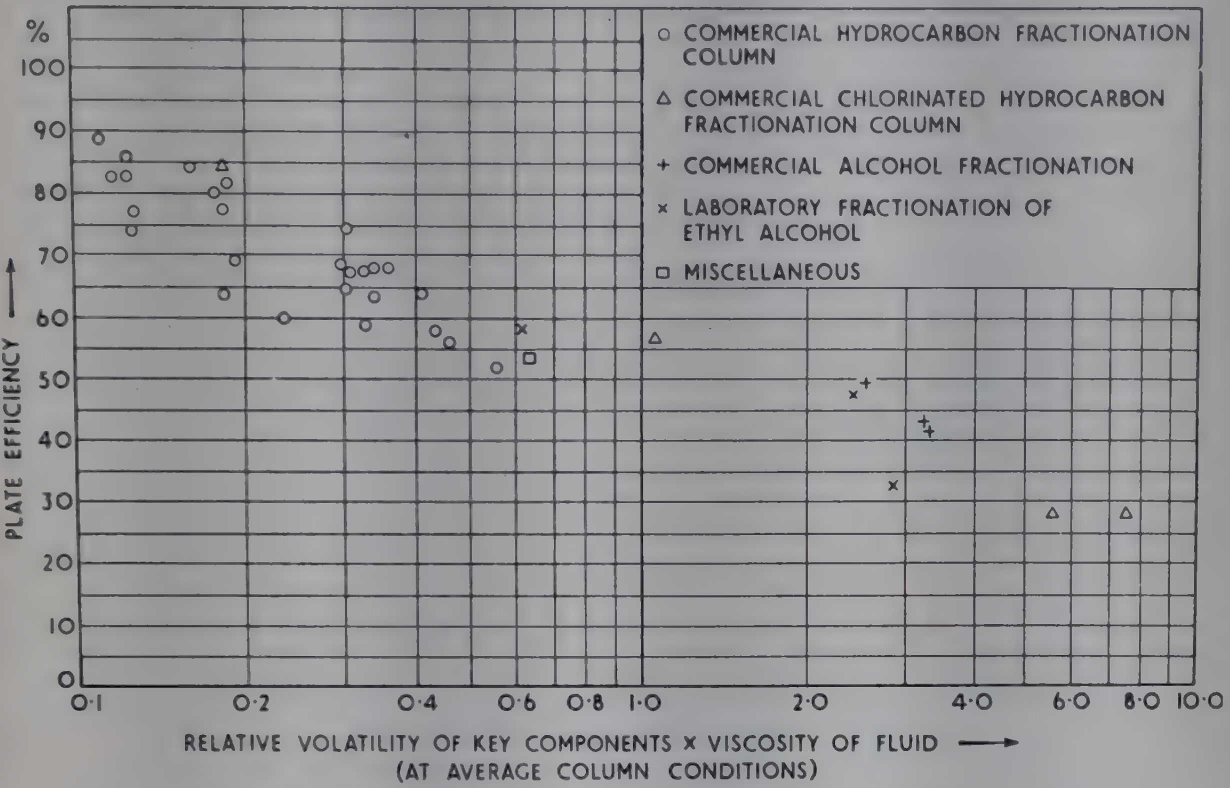


Fig. 18.22. O'CONNELL's relation for plate efficiency

where $L, V$ are liquid and vapour flow rates in lb-mol/hr,

$\mu_L$ is the viscosity of the liquid feed in centipoises,

$\alpha$ is the relative volatility of the key components, and

$h_L$ is the effective submergence in ft, taken as the distance from the top of the slot to the weir lip plus half the slot height.

This relationship introduces the relative flow rates of the liquid and vapour, and a simple attempt is made to allow for the depth of the liquid as this determines the time of contact of the bubbles and liquid. The physical properties are included in the liquid viscosity and relative volatility.

### Principles of Column Design

The main features influencing the performance of the column are:

1. Vapour velocity,
2. Vapour density,
3. Entrainment,
4. Hydraulics of the flow of reflux through the column.

Although in many instances these factors are interrelated, it is convenient to consider their effect on plate efficiency, and then to see how they will determine the mechanical layout of the plate. It will be necessary to establish some relations that will give the plate diameter, the plate spacing, the size and number of caps, the length of the weir and the area of the downcomers for the reflux.

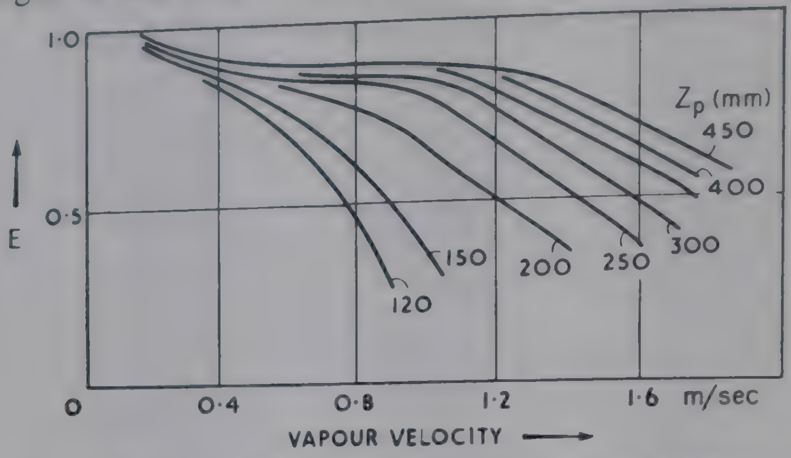


Fig. 18.23. Plate efficiency as a function of vapour velocity for various plate spacings

1. *Influence of Vapour Velocity.* The vapour velocity is the prime factor in determining the diameter of a column, and its effect has been studied by KIRSCHBAUM(4), CAREY(13)(23), and SOUDERS and BROWN(14). KIRSCHBAUM has presented results on a plate 400 mm in diameter with 15 caps; he worked with a mixture of ethyl alcohol and water of 50 mol % concentration, and altered the vapour velocity and the plate spacing. Some of his results are shown in Figs. 18.23, 18.24 and 18.25.

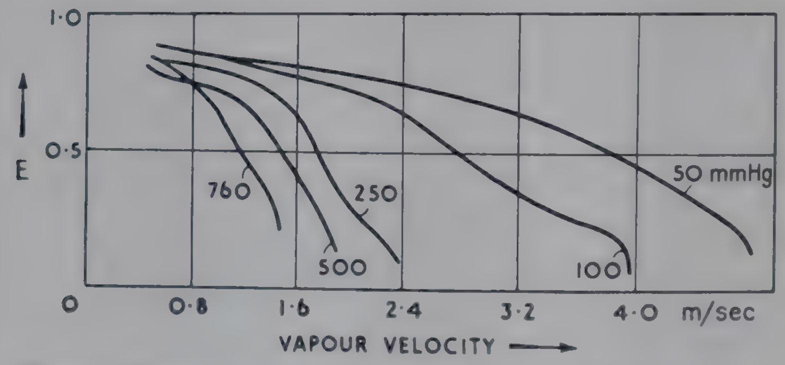


Fig. 18.24. Plate efficiency as a function of vapour velocity for various pressures. Plate spacing 200 mm

Fig. 18.23 shows plate efficiency or enrichment ratio plotted against vapour velocity for various plate spacings ($Z_p$). It may be noted that:—

(*a*) For all plate spacings, $E$ decreases as the velocity increases. This is mainly due to the reduction in contact time between the two phases.

(*b*) The decrease in $E$ is much less with high plate spacings and, with $Z_p > 400$ mm, $E$ remains constant over a wide range of velocities. This arises from an increased rate of transfer and an increased area of contact between phases. There is also much less entrainment of liquid at high spacings.

(*c*) The load limit, or maximum velocity, is determined by the ability of the weirs to carry the reflux liquid. It should be noted that above the liquid on the plate a foam is formed which increases in height with velocity. The pressure drop over the plate rises so rapidly at high velocities that the liquid is prevented from running down.

2. *Influence of Vapour Density.* By operating at reduced pressures, KIRSCHBAUM was able to assess the influence of the vapour density on the enrichment. Fig. 18.24 shows his results for a plate spacing of 200 mm.

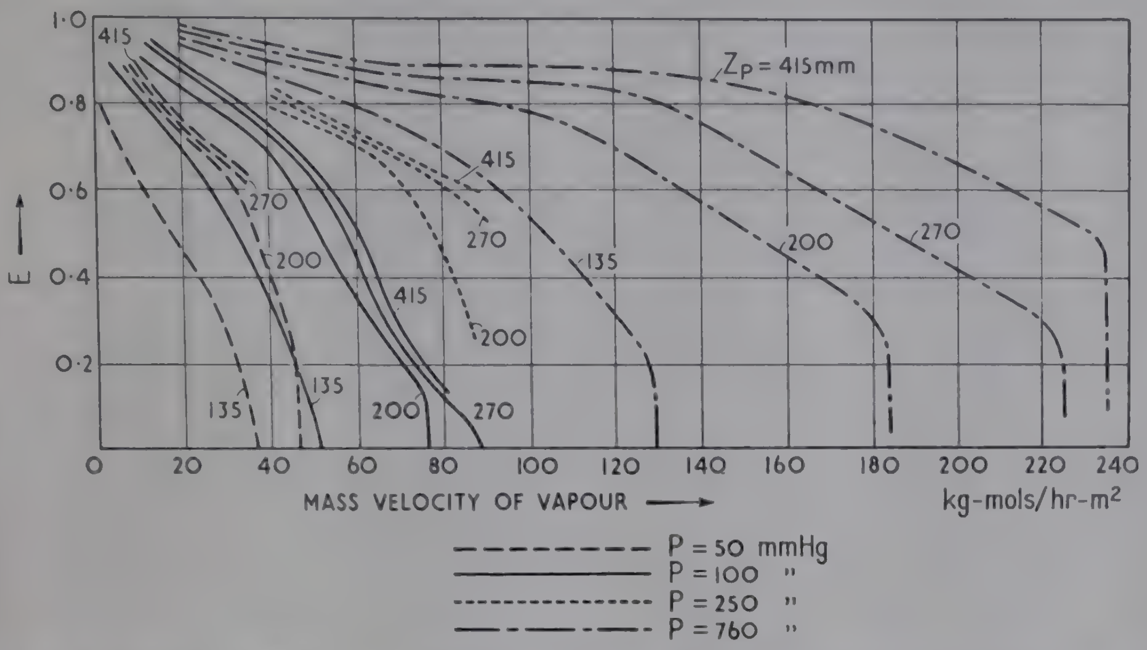


Fig. 18.25. Plate efficiency as a function of mass velocity for various plate spacings (mm) and pressures (mm Hg)

(*a*) At very low vapour velocities, *E* is not affected by pressure. This arises from the fact that there is very little entrainment, and the small differences are attributed to the reduction in foaming tendency at low pressures.

(*b*) For a given value of *E*, much greater velocities are permissible at low pressures.

It is important to note the influence of pressure on the throughput of the column. This is seen by plotting *E* against the mass velocity at different plate spacings and at different pressures. For a given increase in mass velocity, the decrease in *E* is more pronounced the lower the working pressure and the smaller the plate spacing. This is shown by the data in the table below, taken from these curves (Fig. 18.25).

TABLE 18.1. *Effect of throughput on change in working pressure or change in plate efficiency*⁽⁴⁾

| *Plate spacing 200 mm* | | | |
|---|---|---|---|
| *Plate efficiency* | *Total pressure* (*mm* Hg) | *Vapour density* ($Kg/m^3$) | *Throughput* *Kg-mols/hr.* $m^2$ |
| 0·8 | 760 | 1·254 | 92 |
| | 50 | 0·10 | 14 |
| 0·7 | 760 | 1·254 | 120 |
| | 50 | 0·10 | 25 |

Thus, reducing *E* from 0·8 to 0·7 at 760 mm pressure increases the throughput by 28 mols/hr or 33%, but at 50 mm pressure by 13 mols/hr or 55%. Raising the pressure from 50 to 760 mm, with *E* at 0·8, increases the throughput by 67 mols/hr or 268%, whilst if the column is operated with *E* of 0·7 the increase is 82 mols/hr or 215%.

By regarding the upper limit of the throughput as that at which the efficiency falls rapidly, KIRSCHBAUM was able to show that the maximum vapour velocity was related to the vapour density by

$$u = C' . \sqrt{\rho_v} \qquad \ldots .(18.60)$$

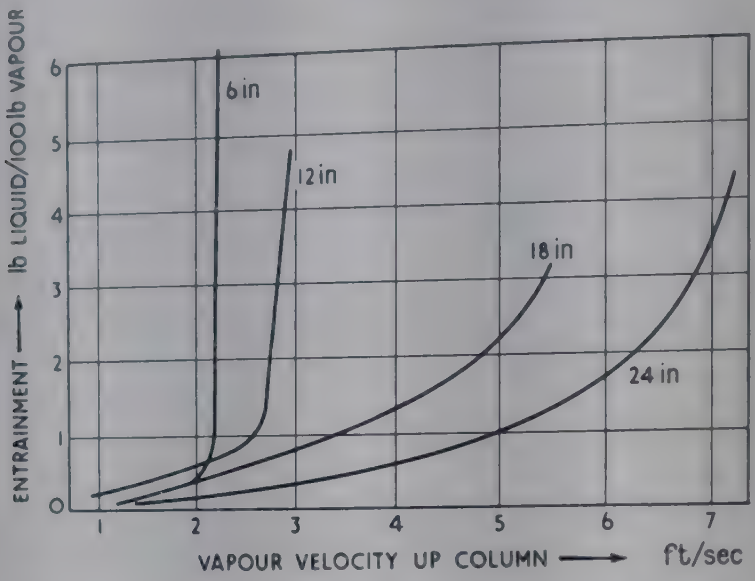


Fig. 18.26. Entrainment as a function of vapour velocity for various plate spacings

where *u* is the vapour velocity in metres per sec,

$\rho_v$ is the vapour density in kg/m³, and

$C'$ varies with plate spacing: $Z_p = 400$ mm, $C' = 1{\cdot}1$

$Z_p = 200$ mm, $C' = 0{\cdot}82$.

3. *Influence of Entrainment on Plate Efficiency.* The extent of entrainment of liquid by the vapour rising over a plate has been studied by a number of workers. This entrainment, expressed as pounds of liquid carried over 100 pounds of vapour, has been found to vary with vapour velocity in the slot and the column and with the plate spacing.

STRANG(15) measured the extent of entrainment in a mild steel column of 3 ft diameter, fitted with 2 plates which could be separated with sections of 6, 12 or 24 in. 27 hexagonal-shaped caps were fitted to each plate, and the height of the overflow was adjustable. Water was supplied to the bottom plate and the overflow passed out of the apparatus. Air was blown in at the bottom and, as it passed up through the first plate, it entrained water, which was collected and measured. He found that the amount of entrainment was very small until a certain vapour velocity was reached, when a further increase in velocity gave a rapid increase in entrainment. The critical velocities, based on the whole cross sectional area of the column, at which there is a sharp increase in entrainment for different plate spacings are given below and are shown in Fig. 18.26.

| Plate Spacing ins. | 6 | 12 | 18 | 24 |
|---|---|---|---|---|
| Critical Velocity ft/sec | 2.1 | 2.7 | 5.1 | 6.7 |

Somewhat similar work has been carried out by Peavy and Baker(21). Colburn(20) has shown that the effect on plate efficiency is not serious until the entrainment exceeds 0·1 mols of liquid per mol of vapour.

***Mechanical Considerations.*** In the previous sections we have seen how the plate efficiency is influenced by vapour velocity, plate spacing and by physical properties of the fluids. The method outlined below for the design of a column is based largely on the work of Carey, Kirkbride, and Bolles. In the first place, a diameter is chosen based on mechanical considerations, such as freedom from entrainment and ability to handle the liquid rates. It is then possible to carry out more detailed design for the plate, from considerations of the permissible vapour capacity of the caps, and liquor capacity of the weirs.

Because small diameter towers are frequently installed inside buildings, the plate spacing is made as small as possible (6–12 in.). For columns larger than 4 ft diameter the minimum spacing is 12 in., and for the large columns used in the petroleum industry 24 in. spacing is commonly used, as this enables a manhole to be fitted to each plate. In some forms of construction it is not necessary to fit manholes, because it is possible to climb up or down inside the tower by removing small sections of the plates.

***Plate Spacing based on Entrainment.*** If we suppose a bubble of liquid of diameter $d$ to be suspended in the vapour stream, then the force downwards due to gravity is balanced by the fluid resistance, so that,

$$\frac{R'}{\rho_v u^2}\frac{\pi . d^2}{4} . \rho_v u^2 = \frac{1}{6} . \pi . d^3(\rho_L - \rho_v)g$$

or

$$u = \left[\frac{2 . gd . (\rho_L - \rho_v)}{3\left(\dfrac{R'}{\rho_v u^2}\right)\rho_v}\right]^{1/2} \qquad \text{....(18.61)}$$

where $\dfrac{R'}{\rho_v u^2}$ is the drag coefficient referred to in Chapter 11.

Thus, we can write

$$u = B\sqrt{\frac{\rho_L - \rho_v}{\rho_v}} \qquad \text{....(18.62)}$$

where

$$B = \left(\frac{2 . g . d}{3 \cdot \dfrac{R'}{\rho_v u^2}}\right)^{1/2}$$

This equation is often expressed in terms of the mass velocity $G'$, as

$$\begin{aligned} G' &= \rho_v u \\ &= B\sqrt{\rho_v(\rho_L - \rho_v)} \qquad \text{....(18.63)} \end{aligned}$$

The value of *B* depends on the plate spacing and liquid seal, some recommended values (in lb/sec-ft$^2$) being given in the Table—

| *Plate spacing in inches* | *Liquid seal in inches* | | | | *Recommended diameter in feet* |
|---|---|---|---|---|---|
| | ½ | 1 | 2 | 3 | |
| 6 | 0·02–0·04 | — | — | — | 2–3 |
| 12 | 0·09–0·11 | 0·07–0·09 | 0·05–0·07 | — | 2–3 |
| 18 | 0·15 | 0·14 | 0·12 | 0·09 | 3–4 |
| 24 | 0·185 | 0·17 | 0·16 | 0·15 | 4–8 |
| 36 | 0·205 | 0·195 | 0·19 | 0·185 | |

For preliminary calculations, a liquid seal of 1 in. is used and the diameter should lie within the range given in the last column. It is believed that these values are somewhat conservative, but lower values of *B* should be used if the liquids are known to be foaming. The liquid seal is taken as the sum of the static submergence and the head over the weir.

***Plate Spacing based on Liquor Flow.*** The size of the downcomer must be such that the liquor does not build up much above half way to the next plate. The liquid head in the downspout is required not only to provide a liquid gradient to cause the liquid to flow across the plate, but also to prevent the rising vapour passing up the downcomer in preference to passing through the caps.

***General Types of Trays.*** In forming a preliminary estimate, the proposals of BOLLES(34) are convenient. Four types of trays are considered, the first three having one pass of liquid and the fourth two passes. The low riser type is used where the vapour load is low.

*General Types of Plates*

| | *Low riser* | *Medium riser* | *High riser* | *Two pass* |
|---|---|---|---|---|
| Riser area as % of tower area | 10 | 12·5 | 15 | 10 |
| Downcomer area as % of tower area | 20 | 14 | 8 | 17·5 |
| Weir length as % of tower diameter | 87 | 79·7 | 68·5 | 84·2 |
| Weir height in. | 2·5 | 2·5 | 2·5 | 2·5 |

For these trays, the height of the weir is taken as 2½ in. and the minimum height under the downcomers as 2½ in. In general, a ½ in. seal may be used for trays up to about 4 ft diameter and a 1 in. seal for trays up to 12 ft.

***Bubble Caps.*** A typical bubble cap plate is illustrated in Fig. 18.27*a* and an enlarged view of a bubble cap is shown in Fig. 18.27*b*. The vapour enters through a riser tube (1) and, at low velocities, leaves through the slots to form a stream of bubbles. Since the main aim is to promote intimate mixing between the vapour and the liquid, narrow slots of $\frac{1}{16}$ to $\frac{3}{16}$ in. are commonly used. To reduce friction losses in the flow of the vapour through the cap, the areas of the riser (1), the reversal section (2), and the annulus (3) are made approximately equal. Caps made from pressed steel are generally preferred to those made from cast iron, since they are much simpler and neater to handle; there is now an increasing tendency to use stainless steel caps. The caps are usually spaced on equilateral triangular centres, the minimum distance between two caps being $1$–$1\frac{1}{2}$ in. A clearance of about 3 in. should be allowed between the last row of caps and the overflow weir, and in no case should a cap be brought nearer the outer wall than $1\frac{1}{2}$ in. The bottom or skirt of the cap is frequently placed $\frac{1}{2}$ in. above the plate, but a greater clearance is desirable both for high liquid flows and for fluids containing solids in suspension. This clearance is reduced to a minimum in the case of towers operating under a vacuum, because the drop in pressure must then be kept to a minimum. The distance between the top of the slot and the top of the weir is known as the static submergence ($h_{ss}$), and determines the distance the bubble travels through the liquid; the static submergence varies normally from $\frac{1}{2}$ to 1 in. When a cap is operating normally, the vapour rushes from the slots as a stream and individual bubbles are no longer formed. The slot velocity is frequently of the order of 10 ft/sec. As indicated earlier, the efficiency of the plate will depend on the size of the bubbles

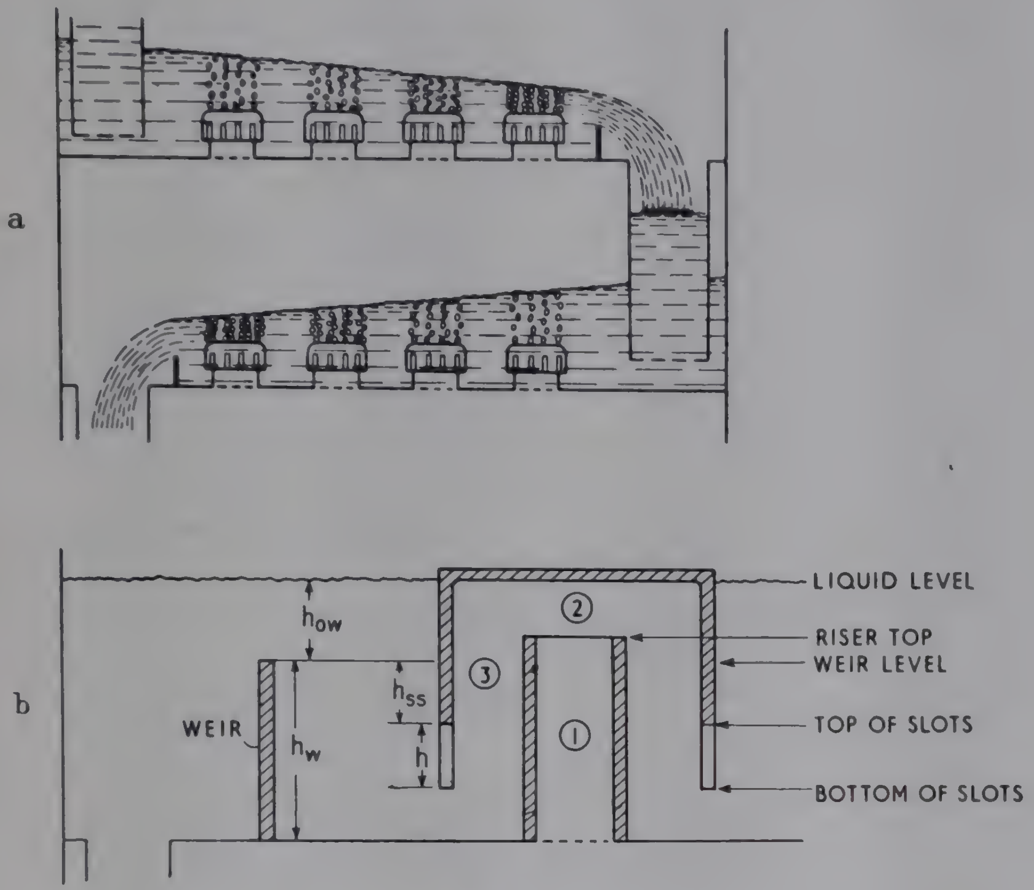


Fig. 18.27
(*a*) Typical bubble cap tray
(*b*) Enlarged view of bubble cap

produced, the depth of liquid through which they pass, the uniformity of the liquor flow over the plate, as well as on the physical properties of the fluids. The geometrical arrangement for three typical caps is indicated in the table below.

*Typical designs for bubble caps*

| | | | | |
|---|---|---|---|---|
| Cap diameter | in. | 3 | 3 | 4 |
| No. of slots | | 38 | 38 | 38 |
| Slot dimensions | in. | $\frac{1}{8} \times \frac{5}{8}$ | $\frac{1}{8} \times 1$ | $\frac{3}{16} \times 1\frac{1}{4}$ |
| Slot area | in$^2$. | 2·97 | 4·75 | 7·75 |
| Approximate riser area | in$^2$. | 3·1 | 3·1 | 6·0 |
| Cap spacing on equilateral triangular centres | in. | 4 | $4\frac{1}{4}$ | $5\frac{3}{4}$ |
| Plate area required per cap | in$^2$. | 13·9 | 15·7 | 28·7 |

***Drop in Pressure through the Cap.*** As the vapour passes through the cap, there is a fall in pressure at the entrance to the risers due to the sudden restriction in area, inside the cap due to friction, and finally in passing out through the slots.

The loss in head $h_r$ due to the contraction of area at the risers is given by

$$h_r = \frac{u_r^2}{2g} \cdot \frac{\rho_v}{\rho_L} \qquad \ldots .(18.64)$$

where $u_r$ is the velocity through the riser.

The loss before the slots can be represented as some multiple of this figure. Taking a value of six, the loss in head in the riser and cap $h_{rc}$ is given by[(5)]

$$h_{rc} = 6 \times h_r$$

$$= 1{\cdot}1\, u_r^2 \cdot \frac{\rho_v}{\rho_L} \quad \text{in inches of liquid} \qquad \ldots .(18.65)$$

The drop in pressure through the slots has been investigated by ROGERS and THIELE[(16)], CROSS and RYDER[(45)], who used an air and water mixture. The slot is regarded as an orifice in which the head varies from zero at the bottom to a maximum at the top. The velocity of the vapour at a distance $z$ above the bottom of the slot is given by

$$C_D \sqrt{2g \,.\, z \left[\frac{\rho_L - \rho_v}{\rho_v}\right]} \qquad \ldots .(18.66)$$

Then the flow of vapour $dv_s$ through a rectangular slot of width $w$, and over a height $dz$ is

$$dv_s = C_D \,.\, w \sqrt{2g \,.\, z \left[\frac{\rho_L - \rho_v}{\rho_v}\right]} \,.\, dz$$

Then total flow through a slot of height $h$,

$$\int_0^{v_s} dv_s = v_s = C_D \,.\, w \int_0^h \sqrt{2g \,.\, z \left[\frac{\rho_L - \rho_v}{\rho_v}\right]} \,.\, dz$$

i.e.

$$v_s = \tfrac{2}{3} \,.\, C_D \,.\, w \sqrt{2g\left(\frac{\rho_L - \rho_v}{\rho_v}\right)} \,.\, h^{3/2} \qquad \ldots.(18.67)$$

$C_D$(16) has a value of approximately 0·51, so that the volumetric capacity $v_s$ of a single slot can be obtained as,

$$v_s = \frac{2}{3} \times 0{\cdot}51 \times \frac{w}{12} \times \sqrt{2g\left(\frac{\rho_L - \rho_v}{\rho_v}\right)} \times \left(\frac{h}{12}\right)^{3/2} \text{ ft}^3/\text{sec}$$

$$= 0{\cdot}0055w \,.\, h^{3/2}\left(\frac{\rho_L - \rho_v}{\rho_v}\right)^{1/2} \qquad \ldots.(18.68)$$

where $w$ and $h$ are expressed in inches.

If $h_s$ is the depth of the slot that is open during the passage of the vapour then we can rearrange equation 18.68 as

$$h_s = 32{\cdot}1\left(\frac{\rho_v}{\rho_L - \rho_v}\right)^{1/3} \,.\, \left(\frac{v_s}{w}\right)^{2/3} \text{ in.} \qquad \ldots.(18.69)$$

***Drop in Pressure over a Plate.*** The vapour in passing through a plate suffers a loss in pressure, first of all in passing through the cap, and then through the depth of liquid above the slots. Expressing this loss in terms of the head of liquid $h_p$ we have

$$h_p = h_{rc} + h_s + h_{ss} + h_{ow} + h_g \qquad \ldots.(18.70)$$

where $h_{ow}$ represents the loss in head over the weir, and $h_g$ the head required to take the liquid across the plate, both measured in inches. Since the liquid on the plate is aerated, the density is less than $\rho_L$, and $0{\cdot}75h_p$ is probably a better approximation for the head lost over plate.

*Liquor Flow.* The depth of liquor at any point on the plate is not easily found. The liquor passing through the downcomer flows over an inlet weir, across the plate, and then over the exit weir. The height of the crest forming over the exit weir $h_{ow}$ can be found by modification of the Francis weir formula. In this case the liquid approaches the weir with an appreciable velocity and the height of the crest will vary with the liquid flow. BOLLES(34) has given a graphical solution for finding $h_{ow}$ from the equation,

$$h_{ow} = 28{\cdot}2F_w\left[\frac{Q'}{w}\right]^{2/3} \qquad \ldots.(18.71)$$

where $h_{ow}$ = height of liquid over weir in inches,

$Q'$ = liquor rate in ft³/sec,

$w$ = weir length in inches, and

$F_w$ = factor as given by BOLLES and taken from Fig. 18.28.

*Hydraulic Gradient.* The liquid at the inlet side of the plate will be at a somewhat higher level above the plate than at the exit weir. This difference in height, which is required to force the liquid across the plate, is denoted by $h_g$, in terms of the head of liquid. A number of workers have measured the hydraulic gradient for various plates using water and air as the fluids. Whilst no entirely satisfactory correlation has been developed, it is possible to get some idea of the value of $h_g$; this should be kept small, preferably not much above 1 in. of water.

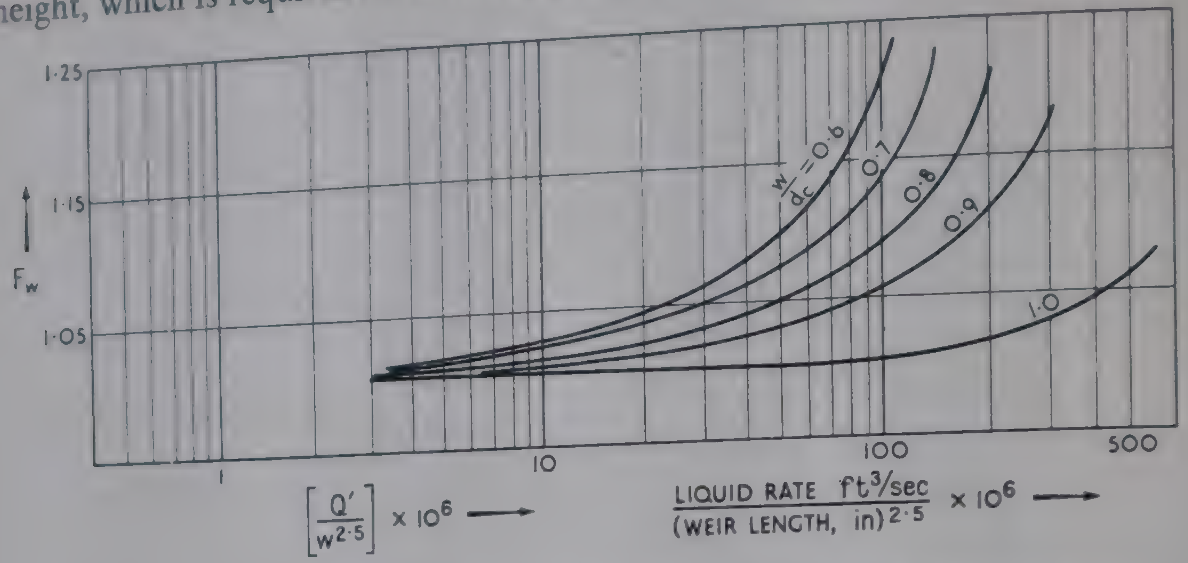


Fig. 18.28. **Bolles'** factor $F_w$ to allow for velocity of approach of liquid at weir (Tower diameter $d_c$ in ft)

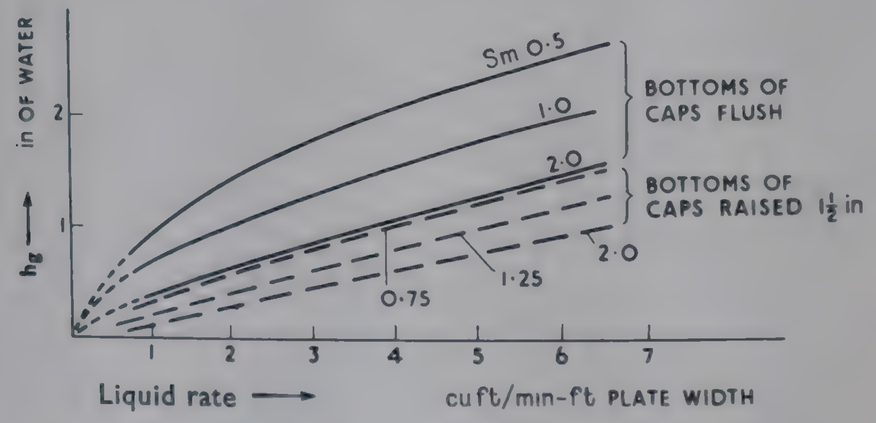


Fig. 18.29. Loss in head over plate as a function of liquid rate—caps on 4½-in. triangular pitch

**Kemp** and **Pyle**[(39)] have used 3 in. and 4 in. caps, and have expressed their results in terms of the group $u_s\sqrt{\rho_v}$, where $u_s$ is the vapour velocity through the slots in ft/sec, and $\rho_v$ is the vapour density in lbs/ft³. For values of $u_s\sqrt{\rho_v} > 10$, there is a danger of the vapour stream from one cap interfering with that from another and giving reduced transfer. They have used various values for the liquid seal $[S_m = (h_{ss} + h_{ow})]$, the depth of liquid above the top of the slots at the exit weir. Some of their results in which the liquid rate is in cu ft/min-ft width of plate are shown in Fig. 18.29. An average value is taken for the liquid rate and $h_g$ is here given in inches of water.

In addition to keeping $h_g$ small, it is essential for the plate to behave in a stable manner. Thus, if the liquid head becomes very high, little vapour will bubble through the row of caps near the inlet weir. The drop in head through the plate at the inlet weir $\Delta h_i$ will be equal to the liquid head above the slots, since no vapour will be passing if the plate is just not stable. The drop in head at the exit weir $\Delta h_e$ will equal the drop in head through the cap at zero seal $\Delta h_o$ together with the liquid head above the slots. These will be approximately equal, so that

$$\Delta h_i = \Delta h_e$$

$$\therefore \qquad h_g + S_m = \Delta h_o + S_m$$

and hence for stable operation $\Delta h_o \geqslant h_g$ ....(18.72)

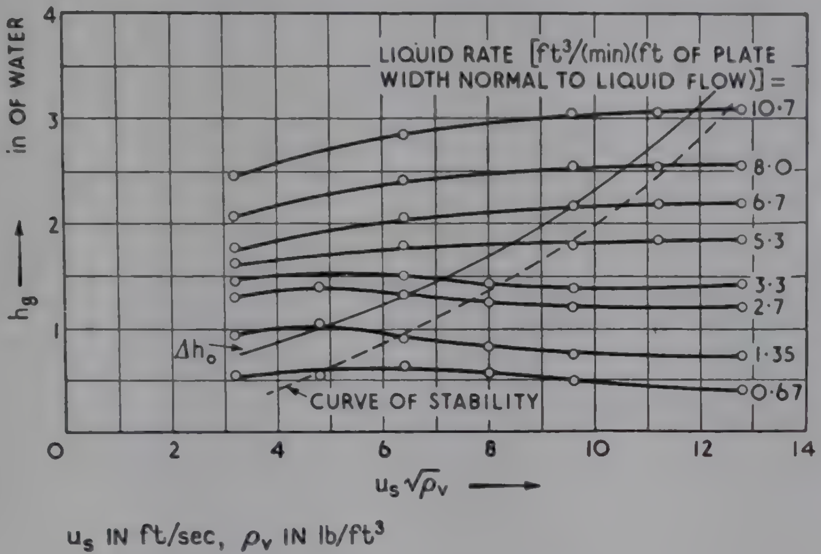


Fig. 18.30. Plate stability

KEMP and PYLE's results for 16 rows of 3 in. caps flush mounted, with a minimum seal of 1 in., are shown in Fig. 18.30, where the full line marked $\Delta h_o$ shows the head drop at zero seal, and the dotted curve, marked "curve of stability," shows the conditions when the first row of caps are just ceasing to pass vapour. Then for stable operation $\Delta h_o$ must be greater than $h_g$. If the values for $u_s\sqrt{\rho_v}$ for various liquid rates are plotted against the number of rows of caps, we get the curves of Fig. 18.31, which show the maximum liquid handling capacity for stable operation. We can now see that to keep $h_g$ at not more than 1 in. of water the liquid rate must not be more than 5½ cu ft/ft-min, unless the minimum seal is increased to a high value. This is why two-pass trays are used for high liquor flows.

GOOD, HUTCHINSON and ROUSSEAU$^{(27)}$ have also presented data, and DAVIES$^{(36)}$ has given a complex correlation of the data.

***Plate Spacing.*** The necessary plate spacing to avoid entrainment of the liquid in the vapour has already been discussed (p. 639). To enable the downcomers to handle the liquor flow, the plate spacing must be such that the liquor does not rise up the downcomer to much above half way between two plates.

The head of liquor in the downcomer $Z_p'$ is made up of the head of liquid on the plate $h_w + h_{ow}$, plus the head lost across the plate $h_p$, plus the head lost as the liquid emerges from the downcomers across the plate $h_d$. This gives

$$Z_p' = h_w + h_{ow} + h_{rc} + h_s + h_{ss} + h_{ow} + h_d + h_g \quad \ldots.(18.73)$$

An approximate relation for this value has been shown by Bolles and Kirkbride to be

$$Z_p' = 2[h_w + h_{ow}] + h_d + h_{rc}$$

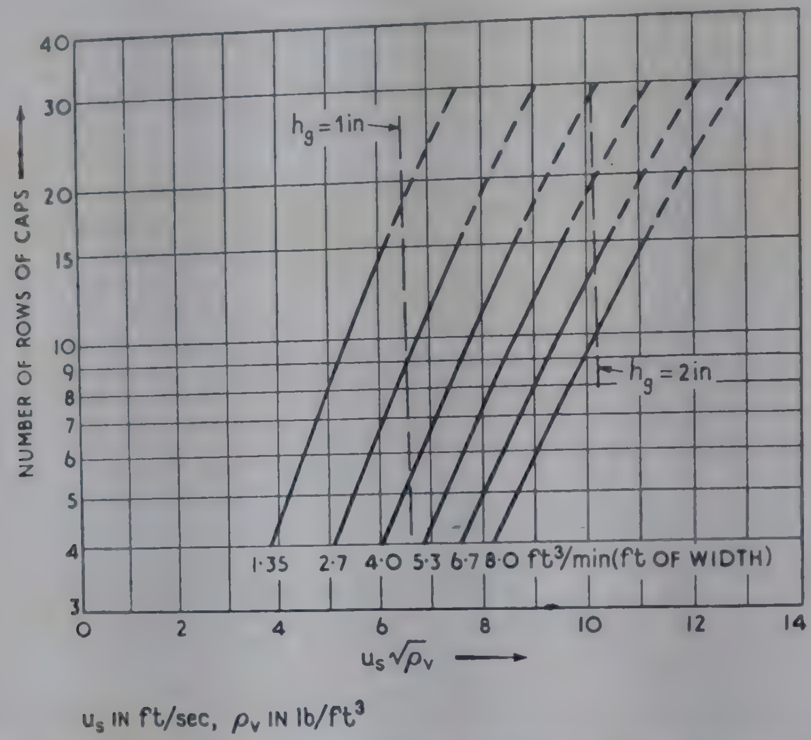
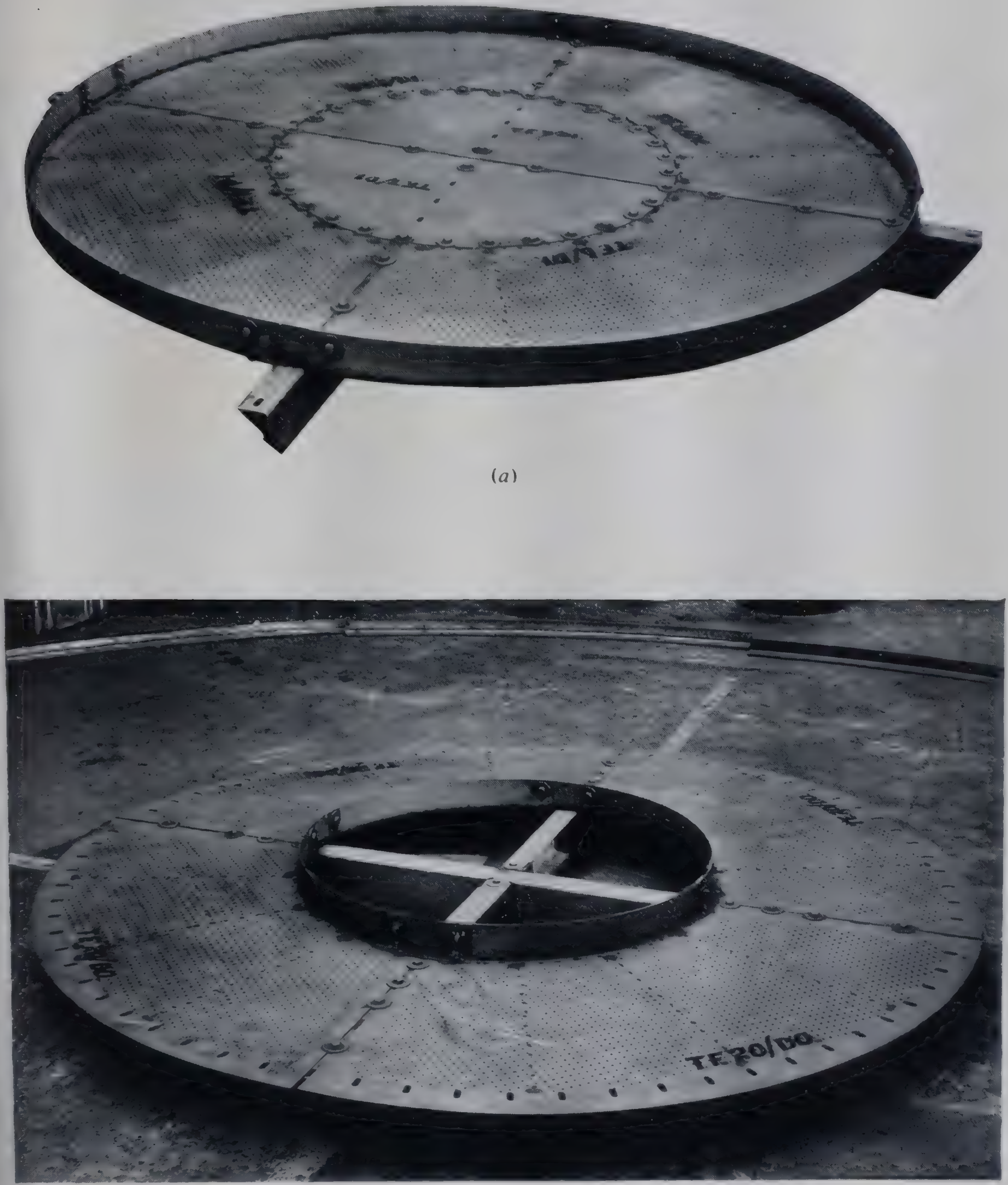


Fig. 18.31. Maximum liquid capacity for stable operation—3 in. caps flush, on $4\frac{1}{4}$-in. triangular pitch, at constant minimum seal of 1 in.

$h_d$ is usually taken as $0{\cdot}6(u_d)^2$, where $u_d$ is the velocity of the liquor in the downcomer in feet per second. The actual velocity in the downcomer may be taken as 0·4 ft/sec, but some workers have suggested that the residence time in the downcomer should be as high as 5 sec. It should be remembered that the liquid on a plate is very much aerated and hence the density of the liquid vapour mixture is substantially less than that of the clear liquid. The plate spacing is frequently made twice the head of liquid in the downcomer.

***Perforated Plate Columns.*** A simple perforated plate may be used as an alternative to the bubble cap plate. The vapour bubbles through a series of small holes of about 2–4 mm in diameter, and the liquid passes down through overflow pipes as with bubble cap columns. These plates give a high efficiency over a rather critical range of vapour flow, but they are much cheaper than the bubble cap types. They are particularly suitable for clear liquids, but any solids in suspension will choke up the small holes rather rapidly.

Some results on a small column of 11 cm diameter with holes of 2 mm diameter on a 7 mm pitch are given in Fig. 18.33, taken from the work of Kirschbaum[4]. At low vapour rates the efficiency is low because the liquid



(a)

(b)

Fig. 18.32. A perforated plate showing disc tray (*a*), and doughnut tray (*b*), for 7 ft 6 in. column

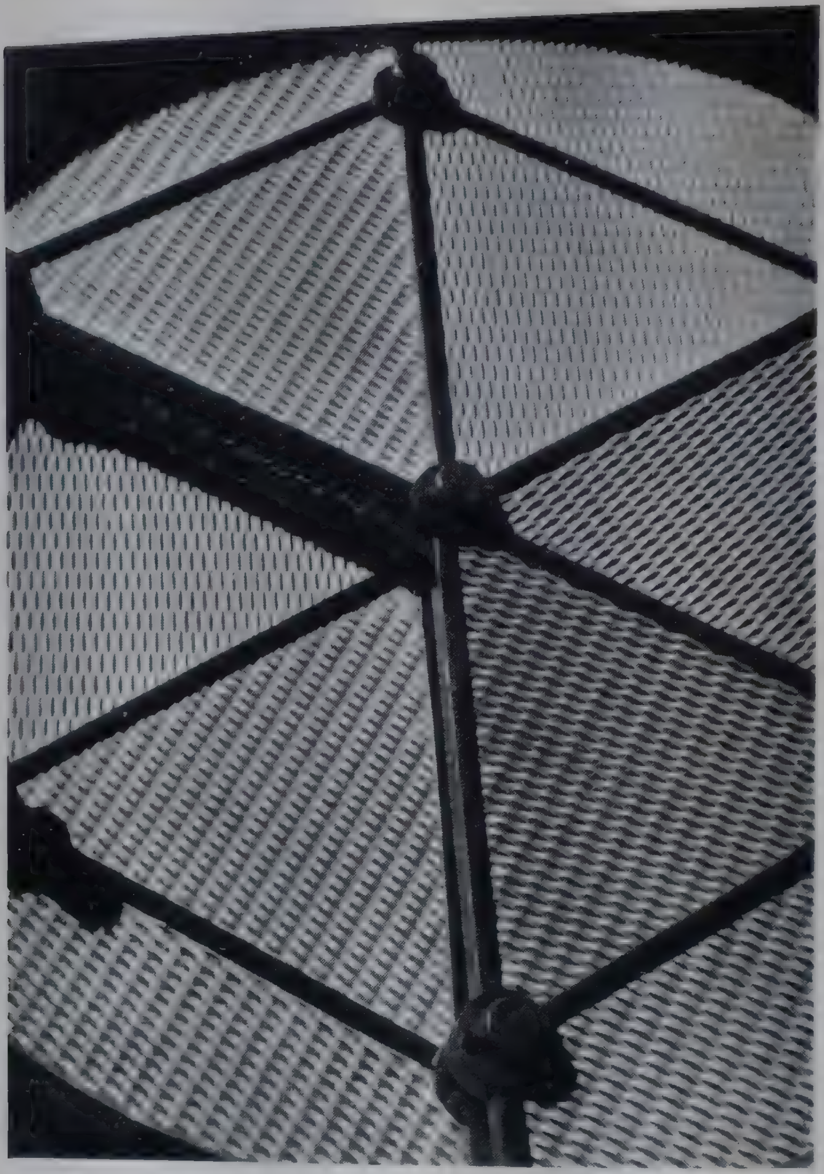


Fig. 18.35. A Kittel plate

drops through the plates. Increasing the vapour rate gives much higher efficiency, but at high rates the liquor is swept off the plates and the efficiency falls. The good foaming action, which is uniform over the plate, gives an excellent medium for contacting the vapour and liquid, and entrainment is avoided with moderate plate spacing. The pressure drop through the plates rises rapidly with vapour velocity, and this may limit the capacity of the plate as compared with bubble caps. It is particularly important with these plates to see that they are horizontal, since slight unevenness will give rise to the liquid passing through the holes.

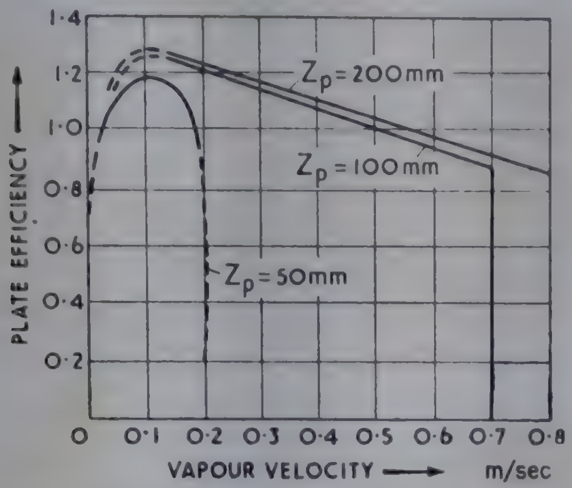


Fig. 18.33. Efficiency of a perforated plate as a function of vapour velocity

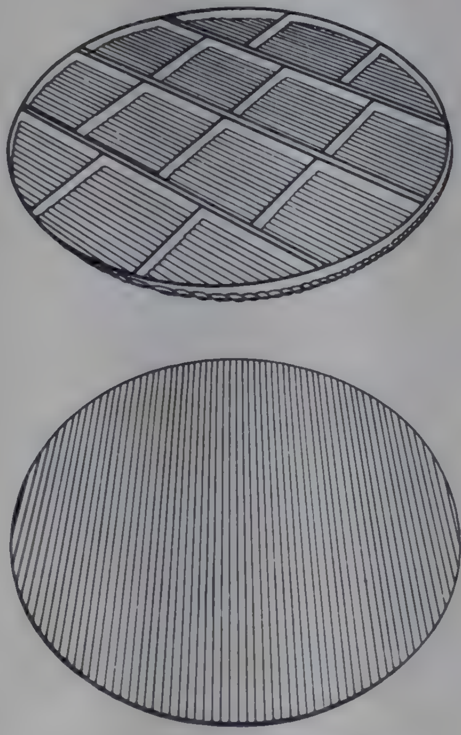


Fig. 18.34. Turbogrid trays

A disc and doughnut arrangement, as shown in Fig. 18.32, is often used with perforated plates.

A new type of tray is the Turbogrid design, one form of which is illustrated in Fig. 18.34. These consist of a flat grid of parallel slots extending over the whole section of the column. The slots may be stamped in a metal plate or, as in the figure, be formed by the spaces between the parallel bars. In normal operation no downcomers are fitted for the liquid, the slots serving both as vapour and liquid channels. The interaction of the vapour and liquid results in liquid building up on the tray, the holdup being determined by the drop in pressure through the plate. It is claimed(48) that an increase in capacity is obtained with these trays as compared with bubble cap trays, and the cost is substantially less.

Another newcomer to this class of equipment is the Kittel plate, shown in Fig. 18.35. This is made of expanded metal, and the vapour passes up through most of the plate, the liquid running down over a small area at the opposite ends.

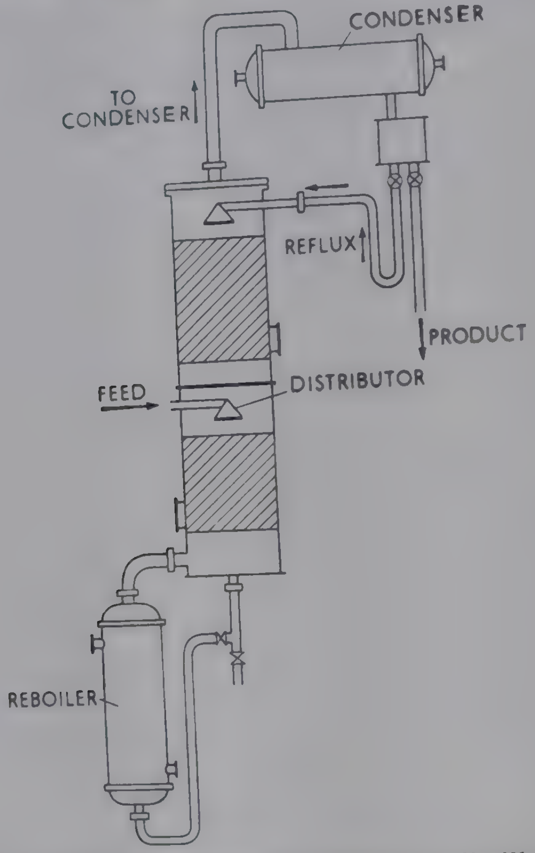


Fig. 18.36. A packed column for distillation

## PACKED COLUMNS FOR DISTILLATION

In the bubble cap and perforated plate columns so far considered, a large interfacial area between the rising vapour and the reflux has been obtained by causing the vapour to bubble through the liquid. An alternative arrangement, which also provides the necessary large interfacial area for diffusion, is the packed column, in which the cylindrical shell of the column is filled with some form of packing. A common arrangement is as indicated in Fig. 18.36, where the packing may consist of Raschig or Lessing rings, Berl saddles, or other shaped particles, all of which are designed to provide a high interfacial area for transfer. These have already been referred to in Chapter 11. In these columns the vapour flows steadily up and the reflux steadily down, giving a true counter-current system in contrast to the conditions in bubble cap columns, where the process of enrichment is stagewise.

## Packings

A selection of suitable packing material is based on the same arguments as for absorption towers (see p. 708), but for industrial units the most usual packings are rings, and the material of construction is determined by the corrosive nature of the fluids. It is important to realise that in a distillation system operating at high reflux rates the weight of reflux is approximately equal to the weight of vapour, but that at low reflux ratios the weight of the liquid is only a small fraction of that of the vapour. Since the vapour has a much lower density than the liquid, the process is really one in which a small quantity of liquid passes through the vapour, and the establishment of good distribution of the liquid is more difficult than in absorption towers, where the two streams are more nearly balanced. The maximum flows of liquid and vapour, which are in practice determined by the boil-up rate, can be calculated by the methods given in Chapter 11, since a column must not operate at or near the flooding point.

## Calculation of Enrichment in Packed Columns

With the plate columns previously discussed, the vapour leaving a plate will be richer in the more volatile component than the vapour entering the plate, by one equilibrium step. PETERS(7) supposed that this same enrichment of the vapour will occur in a certain height of packing, which he denoted by the height equivalent of a theoretical plate (H.E.T.P.). As all sections of the packing are physically the same, it is assumed that one equilibrium stage is represented by a given height of packing. Thus the required height of packing for any desired separation is given by H.E.T.P. × (No. of ideal stages required).

This is a simple method of representation which has been widely used as a method of design. Despite this fact, there have been few developments in the theory. MURCH(46) has given the following relationships for the H.E.T.P. from an analysis of the results of a number of workers. He has considered columns from 2–30 in. diameter and packed over heights of 3–10 ft with rings, saddles, and other packings. Most of the results were for conditions of total reflux, with a vapour rate of 130–1840 lbs/hr-ft² (25–80% of flooding).

$$\text{H.E.T.P.} = C_1 \,.\, G'^{C_2} \,.\, d_c^{C_3} \,.\, Z^{1/3} \,.\, \frac{\alpha \,.\, \mu_L}{\rho_L} \qquad \ldots .(18.74)$$

where the values of $C_1$, $C_2$, $C_3$ varied with packings as given in the Table 18.2.

It should also be noted that the mixtures considered were mainly hydrocarbons with values of relative volatilities not much above three.

In this equation,

$G'$ = mass velocity of vapour in lbs/hr-ft² of tower area,
$d_c$ = column diameter in in.,
$Z$ = packed height in ft.,
$\alpha$ = relative volatility,
$\mu_L$ = liquid viscosity in centipoises, and
$\rho_L$ = liquid density in gm/cc.

TABLE 18.2. *Constants for use in equation 18.74 by* MURCH

| *Type of Packing* | *Size in.* | $C_1$ | $C_2$ | $C_3$ |
|---|---|---|---|---|
| Rings | $\frac{1}{4}$ | | | 1·24 |
| | $\frac{3}{8}$ | 2·10 | −0·37 | 1·24 |
| | $\frac{1}{2}$ | 8·53 | −0·24 | 1·24 |
| | 1 | 0·57 | −0·10 | 1·24 |
| | 2 | 0·42 | 0 | 1·24 |
| Saddles | $\frac{1}{2}$ | 5·62 | −0·45 | 1·11 |
| | 1 | 0·76 | −0·14 | 1·11 |
| Raschig rings of protruded metal | $\frac{1}{4}$ | 0·39 | 0·25 | 0·30 |
| | $\frac{3}{8}$ | 0·076 | 0·50 | 0·30 |
| | $\frac{3}{4}$ | 0·45 | 0·30 | 0·30 |
| | 1 | 3·06 | 0·12 | 0·30 |

ELLIS(47) has presented the following general equation for the H.E.T.P. ($Z_t$) of packed columns using 1–2 in. Raschig rings

$$Z_t = 18d_r + 12m\left[\frac{G'}{L'} - 1\right] \qquad \text{....(18.75)}$$

where $d_r$ is the diameter of the rings,
$m$ is the average slope of equilibrium curve,
$G'$ is the vapour flow rate in lb/hr-ft$^2$,
$L'$ is the liquor flow rate in lb/hr-ft$^2$.

## The Method of Transfer Units

PETERS' proposals are really the application of the stagewise mechanism for the plate column to the packed tower, where however the process is one of countercurrent mass transfer. CHILTON and COLBURN(19) have put forward proposals based on this latter idea of the process. The degree of separation is represented by the rate of change of composition of a component with height of packing, i.e. $\frac{dy}{dZ}$. This rate of change of composition will be dependent upon the equipment, the operating conditions, and on the diffusional potential across the two films. Over the vapour film this driving force is measured by $y_i - y$ or $(\Delta y)_f$ where $y_i$ is the mol fraction of the diffusing component at the interface and $y$ the value for the vapour.

The performance of the column can thus be represented by $E'$, the change in composition with height for unit driving force:

$$E' = \frac{\dfrac{dy}{dZ}}{(\Delta y)_f} \qquad \text{....(18.76)}$$

since, although the efficiency may depend on the equipment and the operating conditions, it should not be influenced by any property of the mixture being separated—in particular by $(\Delta y)_f$.

A relation between $\frac{\mathrm{d}y}{\mathrm{d}Z}$ and $(\Delta y)_f$ can be obtained on the basis of the two-film theory for diffusion (Chapter 7). For the vapour film we have from Fick's law (from equation 7.1).

$$N_A = -D_V \frac{\mathrm{d}C}{\mathrm{d}z} \quad \ldots.(18.77)$$

where $N_A$ is the molal rate of transfer per unit area of interface of component $A$,
$D_V$ is the vapour diffusion coefficient,
$C$ is the concentration in mols per unit volume, and
$z$ is the distance in direction of diffusion.

For an ideal gas, this gives

$$N_A = -\frac{D_V}{\mathbf{R}\,.\,T}\,.\,\frac{\mathrm{d}P_A}{\mathrm{d}z} = -\frac{D_V\,.\,P}{\mathbf{R}\,.\,T}\,.\,\frac{\mathrm{d}y}{\mathrm{d}z} \quad \ldots.(18.78)$$

where $y$ is the mol fraction of $A$.

The negative sign occurs because $z$ is taken in the direction of diffusion from the interface, and $y$ decreases in this direction.

For equimolecular counterdiffusion,

integration gives $$N_A = -\frac{D_V\,.\,P}{\mathbf{R}\,.\,T\,.\,z}\,.\,(\Delta y)_f \quad \ldots.(18.79)$$

or $$N_A = k_g'\,.\,(\Delta y)_f \quad \ldots.(18.80)$$

where $$k_g' = -\frac{D_V\,.\,P}{\mathbf{R}\,.\,T\,.\,z} \quad \ldots.(18.81)$$

Consider a column as indicated in Fig. 18.37, in which the concentration of the more volatile component increases from $y_b$ to $y_t$. Over a small height of the column $\mathrm{d}Z$ the rate of transfer can be written as,

$$k_g'\,.\,a\,.\,s\,.\,(y_i - y)\,\mathrm{d}Z \quad \ldots.(18.82)$$

where $a$ is the active interfacial area for transfer per unit volume of the column, and
$s$ is the cross-sectional area of the column.

But the mols of component $A$ diffusing = total mols of vapour × change in mol fraction

$$= G'\,.\,s\,.\,\mathrm{d}y$$

where $G'$ is the vapour rate in the column in mols/unit time-unit cross section, $k_g'$ is known as the gas-film transfer coefficient, and is measured as mols/unit time-unit area-unit mol fraction difference.

$$G' \,.\, s \,.\, dy = k_g' \,.\, a \,.\, s \,.\, (y_i - y)\, dZ$$

$\therefore$

or

$$E' = \frac{\dfrac{dy}{dZ}}{y_i - y} = \frac{k_g' a}{G'} \qquad \text{from equation 18.76}$$

and

$$\int_{y_b}^{y_t} \frac{dy}{y_i - y} = \frac{k_g' \,.\, a}{G'} \,.\, Z \qquad \ldots.(18.83)$$

where $k_g'a$ is taken as constant over the column.

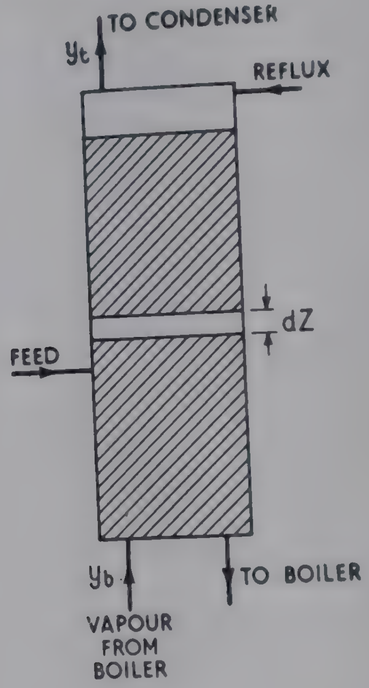


Fig. 18.37. Arrangement for a packed column

The group on the left hand side of this equation represents the integrated ratio of the change in composition to the driving force tending to bring this about. This group has been defined by CHILTON and COLBURN(19) as the number of transfer units $\mathbf{N}_g$, and the quantity $\dfrac{G'}{k_g' \,.\, a}$, which is the reciprocal of the efficiency $E'$ and has the dimensions of length, they define as the height of a transfer unit (H.T.U.) $\mathbf{H}_g$. Equation 18.83 can be written as

$$Z = \mathbf{H}_g \,.\, \mathbf{N}_g \qquad \ldots.(18.84)$$

The concentrations $y_i$, $y$ refer to conditions on either side of the gas film, and hence $\mathbf{N}_g$ is the number of gas-film transfer units, and $\mathbf{H}_g$ the height of a gas-film transfer unit.

For packed columns $\dfrac{k_g'a}{G'}$ represents the point value of the efficiency, and the performance of a packed column is commonly represented by the simple term $\mathbf{H}_g$, a low value of $\mathbf{H}_g$ corresponding to an efficient column. If $\mathbf{H}_g$ is known, the necessary height of a column is found from equation 18.84, since $\mathbf{N}_g$ is determined from the change in concentration required and the shape of the equilibrium curve.

The same number of mols pass through the liquid film and a similar series of equations can be obtained in terms of concentrations across the liquid film.

i.e.
$$G' \,.\, \mathrm{d}y = L' \,.\, \mathrm{d}x \qquad \ldots.(18.85)$$

where $L'$ is the molar flow rate of liquid/unit area, and
$x$ is the mol fraction of the more volatile component in the liquid.

If $k_l'a$ is the mass transfer coefficient for the liquid phase in the mols/unit time-unit volume-unit mol fraction driving force,

then
$$s. L' \,.\, \mathrm{d}x = k_l'as(x - x_i)\, \mathrm{d}Z$$

$$\therefore \qquad \int_{x_b}^{x_t} \frac{\mathrm{d}x}{x - x_i} = \frac{k_l'a}{L'} \,.\, Z \qquad \ldots.(18.86)$$

or
$$\mathbf{N}_l = \frac{1}{\mathbf{H}_l} \,.\, Z$$

where $\mathbf{N}_l$ is the number of liquid film transfer units and
$\mathbf{H}_l$ is the height of a liquid film transfer unit.

***Overall Transfer Coefficients and Transfer Units.*** The driving force over the gas film is taken as $y_i - y$ and over the liquid film as $x - x_i$. If $y_e$ is the concentration in the gas phase in equilibrium with concentration $x$ in the liquid phase, then $y_e - y$ is taken as the overall driving force expressed in terms of $y$. Similarly $x - x_e$ is taken as the overall driving force in terms of $x$, where $x_e$ is the concentration in the liquid in equilibrium with a concentration $y$ in the vapour.

Then the overall driving forces $(\Delta y)_o$ and $(\Delta x)_o$ can be written as

$$(\Delta y)_o = y_e - y$$
$$(\Delta x)_o = x - x_e$$

These overall driving forces can be expressed as the summation of the film driving forces:

$$y_e - y = y_e - y_i + y_i - y$$

and
$$x - x_e = x - x_i + x_i - x_e$$

By analogy with the derivation for film coefficients, we can define a series of overall transfer coefficients and overall transfer units based on these overall driving forces.

Thus number of overall gas transfer units
$$\mathbf{N}_{og} = \int \frac{\mathrm{d}y}{y_e - y} \qquad \ldots.(18.87)$$

and number of overall liquid transfer units
$$\mathbf{N}_{ol} = \int \frac{\mathrm{d}x}{x - x_e} \qquad \ldots.(18.88)$$

The heights of the overall transfer units are
$$\mathbf{H}_{og} = \frac{G'}{K_g' \,.\, a} \qquad \ldots.(18.89)$$

and
$$\mathbf{H}_{ol} = \frac{L'}{K_l' \,.\, a} \qquad \ldots.(18.90)$$

where $K_g' . a$ and $K_l' . a$ are overall transfer coefficients, based on gas or liquid concentrations in mols/unit time-unit volume-unit mol fraction driving force.

***Relation between Overall and Film Transfer Units.*** From the above definitions, we have the following equations:

$$\frac{dZ}{\mathbf{H}_{og}} = \frac{dy}{y_e - y} \qquad \frac{dZ}{\mathbf{H}_g} = \frac{dy}{y_i - y}$$

$$\frac{dZ}{\mathbf{H}_{ol}} = \frac{dx}{x - x_e} \qquad \frac{dZ}{\mathbf{H}_l} = \frac{dx}{x - x_i}$$

$$\therefore \qquad \frac{\mathbf{H}_{og}}{y_e - y} = \frac{\mathbf{H}_g}{y_i - y}$$

and

$$\mathbf{H}_{og} = \mathbf{H}_g \left[\frac{y_e - y_i + y_i - y}{y_i - y}\right] = \mathbf{H}_g \left[1 + \frac{y_e - y_i}{y_i - y}\right]$$

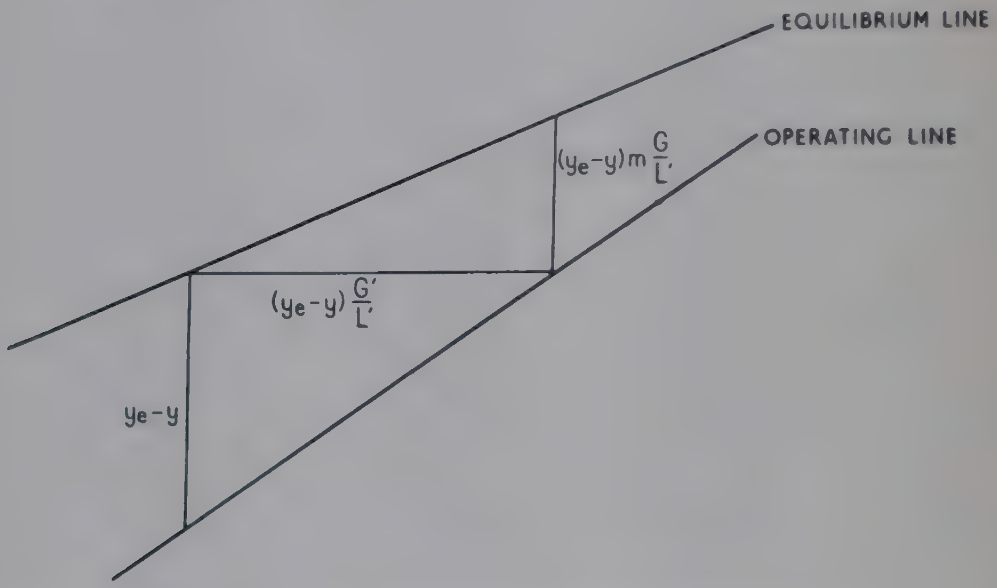


Fig. 18.38. Height equivalent of theoretical plate

If the equilibrium curve is straight over the range $y = y_e$ to $y = y_i$, then assuming equilibrium at the interface,

$$y_e - y_i = m(x - x_i)$$

and

$$\mathbf{H}_{og} = \mathbf{H}_g + m \, . \left(\frac{x - x_i}{y_i - y}\right) \mathbf{H}_g$$

But

$$\frac{x - x_i}{y_i - y} = \frac{dx}{dy} \, . \, \frac{\mathbf{H}_l}{\mathbf{H}_g} = \frac{G'}{L'} \, . \, \frac{\mathbf{H}_l}{\mathbf{H}_g}$$

since $G' \, dy = L' \, dx$.

$$\therefore \qquad \mathbf{H}_{og} = \mathbf{H}_g + \frac{m \, . \, G'}{L'} \, . \, \mathbf{H}_l \qquad \ldots .(18.91)$$

We can see that this form of relationship can be written in terms of transfer coefficients. (See Chapter 19, p. 706)

$$\frac{1}{K_g' \, . \, a} = \frac{1}{k_g' \, . \, a} + \frac{m}{k_l' \, . \, a} \qquad \ldots .(18.92)$$

***Relation of H.T.U. to H.E.T.P.*** In a theoretical plate, the mol fraction of the M.V.C. in the vapour will increase from $y$ to $y_e$, so that the total mass transfer is $G'(y_e - y)$. If the equilibrium curve can be considered straight over the height ($Z_t$) of column equivalent to a theoretical plate, we may use the logarithmic mean driving force. Thus, referring to Fig. 18.38, we have

$$G'(y_e - y) = K_g'a \,.\, Z_t\,(y_e - y)\,\frac{m\dfrac{G'}{L'} - 1}{\ln m\dfrac{G'}{L'}}$$

$$\therefore \quad Z_t = \frac{G'}{K_g'a} \cdot \frac{\ln m\dfrac{G'}{L'}}{m\dfrac{G'}{L'} - 1} = \mathbf{H}_{og} \cdot \frac{\ln m\dfrac{G'}{L'}}{m\dfrac{G'}{L'} - 1} = \mathbf{H}_{og}\,\frac{\ln\left[1 - \left(1 - \dfrac{mG'}{L'}\right)\right]}{m\dfrac{G'}{L'} - 1}$$

$$= \mathbf{H}_{og}\left[1 + \frac{1}{2}\left(1 - m\frac{G'}{L'}\right) + \frac{1}{3}\left(1 - \frac{mG'}{L'}\right)^2 + \dots\right]. \qquad \dots.(18.93)$$

If the operating and equilibrium lines are parallel, $\dfrac{mG'}{L'} = 1$,

and $$Z_t = \mathbf{H}_{og} \qquad \dots.(18.93a)$$

***Determination of Transfer Units (Experimental).*** DUNCAN *et al.*[26], have studied the influence of operating variables on the values of $\mathbf{H}_g$, $\mathbf{H}_{og}$ using a column of 4·81 in. i.d. by 12 ft high. They worked with the mixtures carbon tetrachloride-benzene[(E)], trichlorethylene-toluene[(D)],

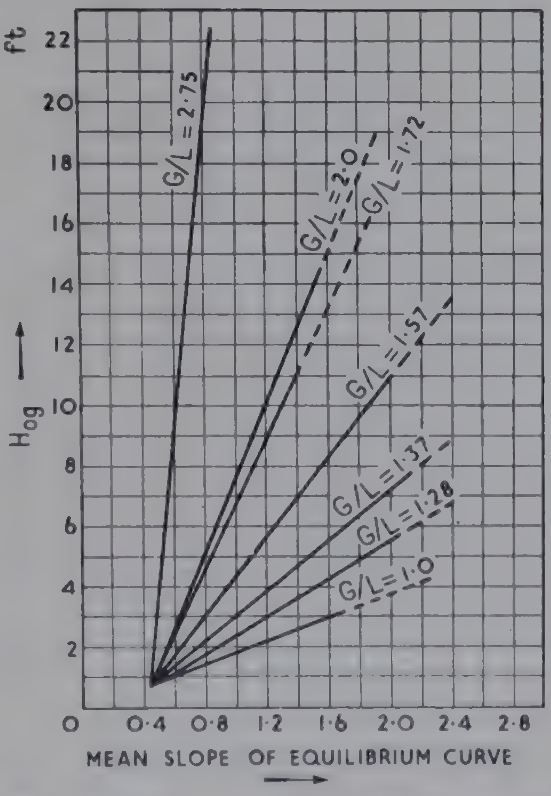


Fig. 18.39. Dependence of the height of the transfer unit on the slope of the equilibrium curve (DUNCAN *et al.*)

methanol-water(B), acetone-water(A), and ethanol-water(C). With some of these systems $m$ varies considerably with concentration; with system E, $m \simeq 1$; with system D, $m$ varies from 2 to $\frac{1}{2}$; whilst with system C, $m$ varies from 13 to $\frac{1}{2}$. Some of their results for methanol-water are shown in Fig. 18.39, where $\mathbf{H}_{og}$ is plotted versus $m$, the average slope of the equilibrium curve, for various values of $G/L$ which are here given as lb/hr-ft², not as mols.

A similar correlation is given by SMITH(49) using a 1 in. glass column operated at total reflux with small and large helices. This diagram (Fig. 18.40) also shows the influence of pressure. Fig. 18.41, also taken from SMITH's work, shows the importance of concentration on the values of $\mathbf{H}_{og}$.

Some general indication of the values of $\mathbf{H}_{og}$ are given in Table 18.3 below, which gives values obtained by FURNAS and TAYLOR(24a) for experiments with ethanol-water mixtures at atmospheric pressure in a column 12 in. diameter operating at total reflux.

TABLE 18.3. *Values of the height of the transfer unit* $\mathbf{H}_{og}$

| *Packing* | *Depth of packing (ft)* | *Liquid rate (lb/hr-sq ft)* | $\mathbf{H}_{og}$ *(ft)* |
|---|---|---|---|
| 2 in. Raschig rings | 10 | 816 | 2·2 |
| 1 in. Raschig rings | 10 | 785 | 1·2 |
| 1 in. Berl saddles | 9 | 150 | 1·4 |
| 1 in. Berl saddles | 9 | 960 | 1·1 |
| ½ in. Berl saddles | 10 | 190 | 1·5 |
| ½ in. Berl saddles | 10 | 920 | 0·9 |
| ⅜ in. Raschig rings | 8 | 320 | 1·3 |
| ⅜ in. Raschig rings | 8 | 600 | 1·0 |

## *Values of H.T.U. in terms of Flow Rates and Physical Properties*

### *Wetted-Wall Columns*

The proposals that have been made for calculating transfer coefficients from physical data of the system and the operating liquid and vapour rates are really all related to conditions existing in a simpler unit in the form of a wetted-wall column. In the wetted-wall column (see Chapter 19), vapour rising from the boiler passes up the column which is lagged to prevent heat loss. The liquid flows down the walls, and it thus provides the simplest form of equipment giving countercurrent flow. The mass transfer in the unit can be expressed by means of the *j*-factor of CHILTON and COLBURN which has already been discussed in Chapter 8.

$$j_d = \frac{k_g'}{G'}\left(\frac{\mu}{\rho D}\right)_v^{2/3} = 0{\cdot}023\left(\frac{d_c u \rho}{\mu}\right)_v^{-0{\cdot}17} \qquad \ldots.(18.94)$$

where the properties refer to the vapour. This type of unit has been studied by GILLILAND and SHERWOOD(17), CHARI and STORROW(43a), SUROWIEC and FURNAS(28) and others.

For a wetted wall column we can write $\dfrac{\text{area of interface}}{\text{volume of column}} = \dfrac{4}{d_c} = a$. We then have

$$\mathbf{H}_g = \frac{G'}{k_g' \,.\, a} = 10{\cdot}9 \,.\, d_c \, Re._g^{0{\cdot}17} \,.\, Sc._g^{2/3} \qquad \ldots.(18.95)$$

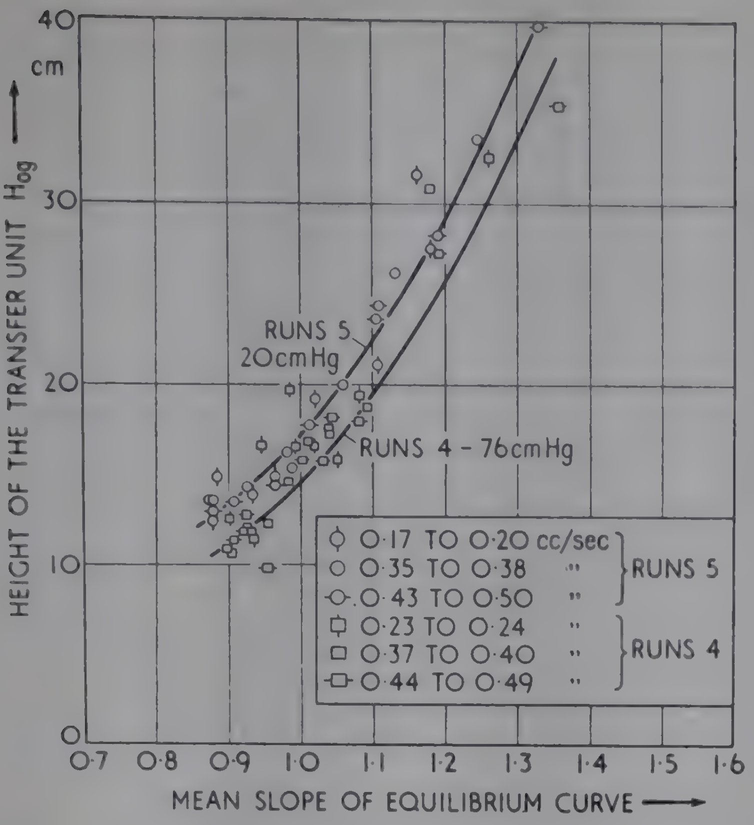


Fig. 18.40. Dependence of the height of the transfer unit on the slope of the equilibrium curve (SMITH)

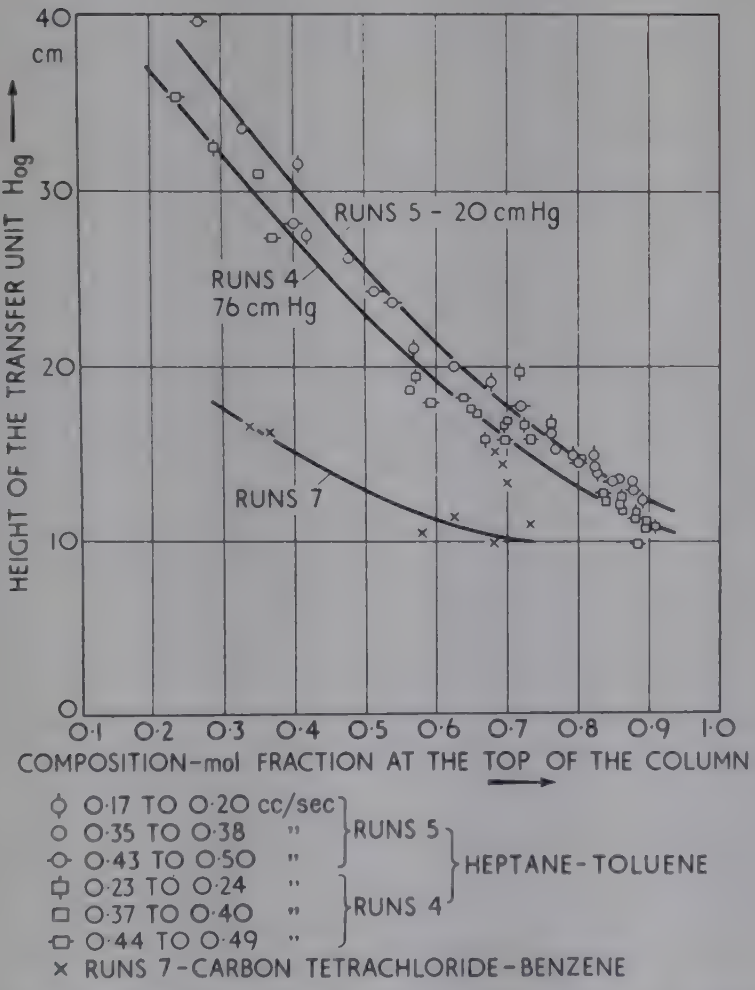


Fig. 18.41. Dependence of the height of the transfer unit on concentration (SMITH)

where the linear characteristic length is taken as the diameter of the column $d_o$. $Re._g$ and $Sc._g$ are the Reynolds and Schmidt Numbers with respect to the vapour. SUROWIEC and FURNAS were able to express their results, obtained with alcohol and water, in this form. For transfer through the liquid film, they derived an expression based on the analysis for heat transfer from a tube to a liquid flowing under viscous conditions down the inside of the tube.

They presented their equation as

$$\mathbf{H}_l = B' \,.\, Z \,.\, \frac{M}{M_m} \,.\, Re._l^{8/9} \,.\, Sc._l^{10/9} \,.\, \left(\frac{D_L^2}{g \,.\, Z^3}\right)^{2/9} \qquad \ldots.(18.96)$$

where $B'$ is a constant,
$M_m$ is the mean molecular weight of the liquid,
$M$ is the point value of the molecular weight,
$Z$ is the height of the tube, and
$Re._l$ and $Sc._l$ are the Reynolds and Schmidt Numbers with respect to the liquid.

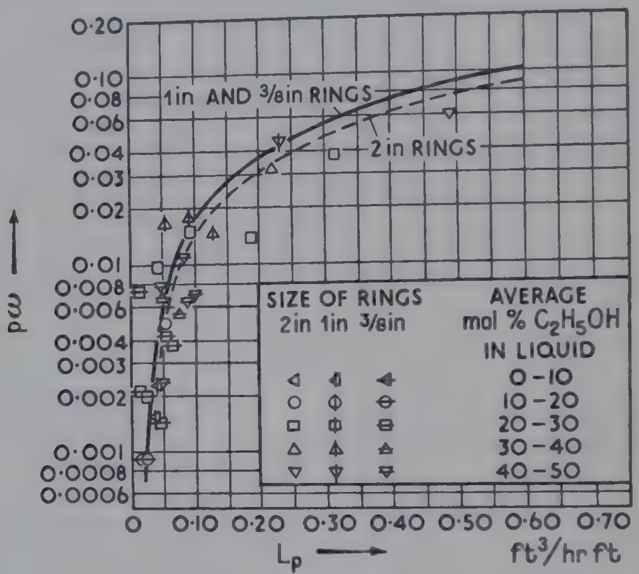


Fig. 18.42. Effect of liquid rate on degree of wetting of packing (PRATT)

COLBURN, however, has suggested that for mass transfer, the transfer in the liquid phase is from a vapour-liquid interface where the liquid velocity is a maximum to the wall where the liquid velocity is zero. With a liquid flowing inside the tube the heat transfer is from a layer of zero velocity at the wall to the fluid at the centre of the tube which is moving with a maximum velocity. HATTA(14a) based his analysis on the more closely related process of diffusion of a gas into a liquid, and obtained the expression

$$\mathbf{H}_l = B'' \,.\, Z \,.\, \frac{M}{M_m} \, Re._l^{0\cdot 67} \,.\, Sc._l^{5/6} \,.\, \left(\frac{D_L^2}{g \,.\, Z^3}\right)^{1/6} \qquad \ldots.(18.97)$$

It will be seen that, despite the difference in the arguments, the two equations are really of a similar nature.

### *Packed Columns*

The application of the ideas indicated above for wetted wall columns to the more complex case of packed columns requires the assumptions: (1) that the mechanism is unchanged and (2) that the expressions are valid over the much wider ranges of flow used in packed columns. This has been attempted by SMITH(49) and PRATT(43).

PRATT(43), starting from the basic equation

$$j_d = \frac{k_g'}{G'}\left(\frac{\mu}{\rho D}\right)_v^{2/3} = \text{const } Re.^{-0\cdot 2} \qquad \ldots.(18.98)$$

has suggested, from an examination of the available data, that the importance of the degree of wetting can be taken into account by writing this in the form,

$$\frac{k_g}{G'} \cdot e \cdot \left(\frac{\mu}{\rho D}\right)_v^{2/3} = p \cdot \omega \cdot \left(\frac{d_e G'}{\mu e}\right)^{-0\cdot25} \qquad \ldots.(18.99)$$

where $G'$ is the mass velocity (mass rate per unit area),
$d_e$ is the hydraulic mean diameter for the packing,
$p$ is a constant,
$\omega$ is the fraction of the packing wetted, and
$e$ is the fractional voidage of the packing.

He gives several plots of $p\omega$ *vs* $L_p$, as indicated in Fig. 18.42, where $L_p$ is the liquid rate based on the periphery of the packing, in cu ft/hr-ft. The periphery is taken as equal to $a$(ft$^2$/ft$^3$), although this is only correct for geometrical systems such as stacked rings.

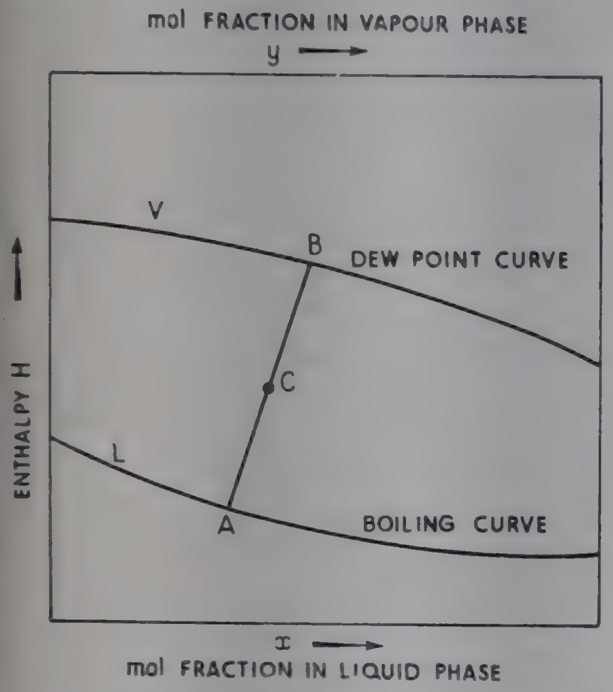


Fig. 18.43. Enthalpy-composition diagram, showing enthalpies of liquid and vapour

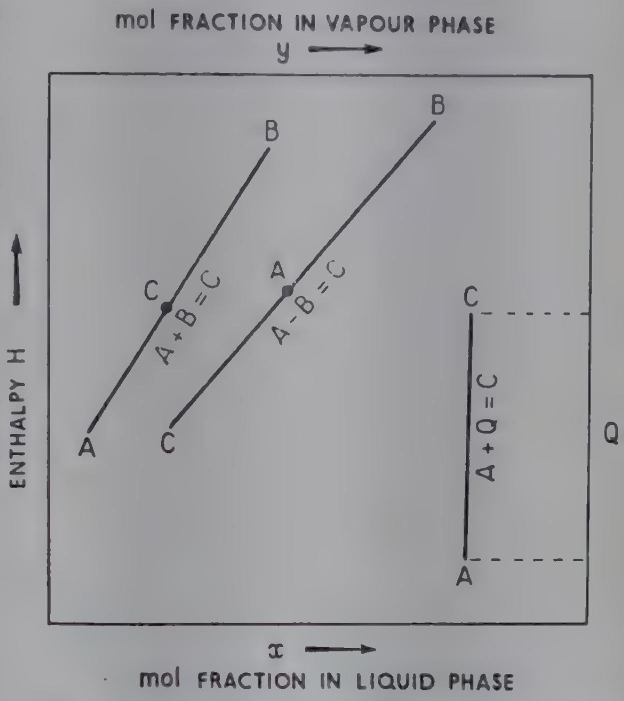


Fig. 18.44. Combination and separation of mixture on enthalpy-composition diagram

## CONDITIONS FOR VARYING OVERFLOW NON-IDEAL BINARY SYSTEMS

In the previous sections we have considered the case of constant molar latent heat, no heat of mixing, and hence constant molar rate of reflux in the column. These simplifying assumptions are extremely useful in that they enable a simple geometrical method to be used for finding the change in concentration on the plates and, whilst they are rarely entirely true in industrial conditions, they often provide a convenient start for design purposes. For a non-ideal system, where the molar latent heat is no longer constant and there is a substantial heat of mixing, the calculations become much more tedious. For binary mixtures of this kind a graphical model has been developed by RUHEMANN(37), PONCHON(6), and SAVARIT(2a), based on the use of an enthalpy-composition chart. A typical enthalpy composition or $H-x$ chart is indicated in Fig. 18.43, where the upper curve $V$ is the dew-point curve, and the lower curve $L$ the boiling point curve. The use of this diagram is based on the following geometrical properties, as illustrated in Fig. 18.44. A quantity of mixture in any physical state is known as a "phase" and is denoted by mass, composition and enthalpy. The phase is indicated upon the diagram by a point which shows enthalpy and composition, but which does not show the mass. If $m$ is the mass, $x$ the composition and $H$ the enthalpy of the phase, then the addition of two phases $A$ and $B$ to give phase $C$ is governed by

$$m_A + m_B = m_C; \qquad \ldots.(18.100a)$$

$$m_A x_A + m_B x_B = m_C x_C; \qquad \ldots.(18.100b)$$

$$m_A H_A + m_B H_B = m_C H_C \qquad \ldots.(18.100c)$$

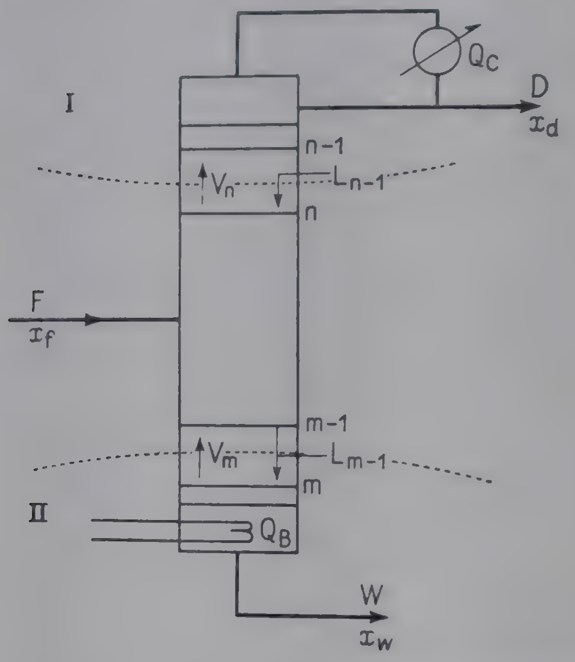


Fig. 18.45. Continuous distillation column

Similarly, a phase $C$ can be regarded as the sum of the phase $A$ and a quantity of heat $Q$, i.e.

$$A + Q = C. \qquad \ldots.(18.101)$$

If $m_A = m_C$ and $x_A = x_C$, then $H_A + \dfrac{Q}{m_A} = H_C$.

Thus the addition of two phases $A$ and $B$ is shown on the diagram by point $C$ on the straight line joining the two phases, whilst the difference $A - B$ is found by a point $C$ on the extension of the line $AB$. If, as shown in Fig. 18.43, we consider a phase represented by $C$ in the region between the dew-point and boiling point curves, then this phase will divide into two phases $A$ and $B$ at the ends of a tie line through the point $C$, so that

$$\frac{m_A}{m_B} = \frac{CB}{CA} \qquad \ldots.(18.102)$$

The $H$-$x$ chart, therefore, enables us to determine geometrically the effect of adding two phases with or without the addition of heat. The diagram may be drawn for unit mass or for one mol of material, but since we are no longer concerned with constant molar reflux it is more convenient to use unit mass as the basis. Thus we shall work for unit mass of product, and calculate the mass of the individual streams as proportions of the product.

Fig. 18.45 represents a continuous distillation unit operating with feed $F$

of composition $x_f$, and giving a top product $D$ of composition $x_d$ and a bottom product $W$ of composition $x_w$. In this analysis, the quantities in the streams $V$ of rising vapour and $L$ of reflux are given in mass units, such as lb/hr, and the composition of the streams as mass fractions, $x$ referring to the liquid and $y$ to the vapour streams.

The plates are numbered from the top downwards, subscript $n$ indicating the rectifying and $m$ the stripping section.

$H^V$ and $H^L$ represent the enthalpy of a vapour and liquid stream respectively.

$Q_C$ is the heat removed in the condenser. (In this case no cooling of product considered.)

$Q_B$ is the heat put into the boiler.

Then the following relationships are obtained by taking material and heat balances above plate $n$:

$$V_n = L_{n-1} + D$$

or

$$V_n - L_{n-1} = D \qquad \ldots.(18.103)$$

$$V_n \cdot y_n = L_{n-1} \cdot x_{n-1} + D \cdot x_d$$

or

$$V_n \cdot y_n - L_{n-1} \cdot x_{n-1} = D \cdot x_d \qquad \ldots.(18.104)$$

$$V_n \cdot H_n^V = L_{n-1} \cdot H_{n-1}^L + D \cdot H_d^L + Q_C$$

or

$$V_n \cdot H_n^V - L_{n-1} \cdot H_{n-1}^L = D \cdot H_d^L + Q_C \qquad \ldots.(18.105)$$

Putting $H_d' = H_d^L + \dfrac{Q_C}{D}$, we can write equation 18.105 as

$$V_n \cdot H_n^V = L_{n-1} \cdot H_{n-1}^L + D \cdot H_d'$$

or

$$V_n \cdot H_n^V - L_{n-1} \cdot H_{n-1}^L = D \cdot H_d' \qquad \ldots.(18.106)$$

Then from equations 18.103 and 18.104,

$$\frac{L_{n-1}}{D} = \frac{x_d - y_n}{y_n - x_{n-1}} \qquad \ldots.(18.107)$$

and from equations 18.103 and 18.106,

$$\frac{L_{n-1}}{D} = \frac{H_d' - H_n^V}{H_n^V - H_{n-1}^L} \qquad \ldots.(18.108)$$

whence

$$\frac{H_d' - H_n^V}{H_n^V - H_{n-1}^L} = \frac{x_d - y_n}{y_n - x_{n-1}} \qquad \ldots.(18.109)$$

or

$$y_n = \left[\frac{H_d' - H_n^V}{H_d' - H_{n-1}^L}\right] \cdot x_{n-1} + \left[\frac{H_n^V - H_{n-1}^L}{H_d' - H_{n-1}^L}\right] \cdot x_d \qquad \ldots.(18.110)$$

This equation (18.110) is that of any operating line relating the composition of the vapour $y_n$ rising from a plate to the composition of the liquid reflux entering the plate; or alternatively it represents the relation between the composition of the vapour and liquid streams between any two plates. From equation 18.109, we can see that all such operating lines pass through a common pole $N$ of co-ordinates $x_d$ and $H_d'$.

Alternatively, noting that the right hand side of equations 18.103, 104, 105, are independent of conditions below the feed plate, we can define a stream $N$ of mass equal to the difference between the vapour and liquid streams between two plates, of composition $x_d$ and of enthalpy $H_d'$. Then the three quantities $V_n$, $L_{n-1}$ and $N$ will be on a straight line passing through $N$, as shown in Fig. 18.46.

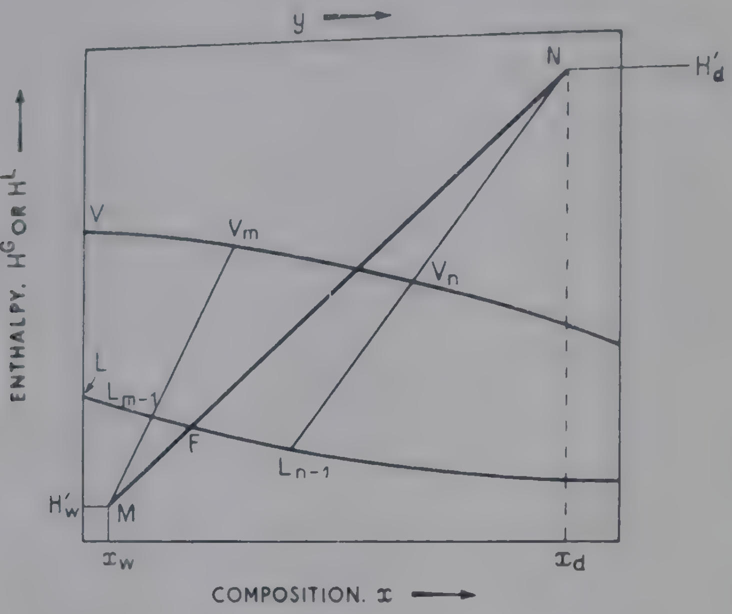


Fig. 18.46. Enthalpy-composition diagram

Below the feed plate we can write a similar series of equations for material and heat balances.

$$V_m + W = L_{m-1}$$

or

$$-V_m + L_{m-1} = W \qquad \text{....(18.111)}$$

$$V_m \cdot y_m + W \cdot x_w = L_{m-1} \cdot x_{m-1}$$

or

$$-V_m \cdot y_m + L_{m-1} \cdot x_{m-1} = W \cdot x_w \qquad \text{....(18.112)}$$

$$V_m \cdot H_m^V + W \cdot H_w{}^L = L_{m-1} \cdot H_{m-1}^L + Q_B$$

or

$$-V_m \cdot H_m^V + L_{m-1} \cdot H_{m-1}^L = W \cdot H_w^L - Q_B \qquad \text{....(18.113)}$$

And putting

$$H_w' = H_w^L - \frac{Q_B}{W} \qquad \text{....(18.114)}$$

$$-V \cdot H_m^V + L_{m-1} \cdot H_{m-1}^L = W \cdot H_w' \qquad \text{....(18.115)}$$

Then

$$\frac{L_{m-1}}{W} = \frac{-x_w + y_m}{y_m - x_{m-1}} \qquad \text{....(18.116)}$$

and

$$\frac{L_{m-1}}{W} = \frac{-H_w' + H_m^V}{H_m^V - H_{m-1}^L} \qquad \text{....(18.117)}$$

whence

$$\frac{-H_w' + H_m^V}{H_m^V - H_{m-1}^L} = \frac{-x_w + y_m}{y_m - x_{m-1}} \qquad \text{....(18.118)}$$

This equation represents any operating line below the feed plate, and shows that all such lines pass through a common pole $M$ of co-ordinates $x_w$ and $H_w'$. As with the rectifying section, we can define a stream $M$ of mass $V_m - V_{m-1}$ and of composition $x_w$ and enthalpy $H_w'$.

Then $$F = M + N \qquad \text{....(18.119)}$$

and $$F \,.\, x_f = M \,.\, x_w + N \,.\, x_d \qquad \text{....(18.120)}$$

It thus follows that phases $F$, $M$, and $N$ will be on a straight line on the $H$-$x$ chart, as indicated in Figs. 18.46 and 18.47.

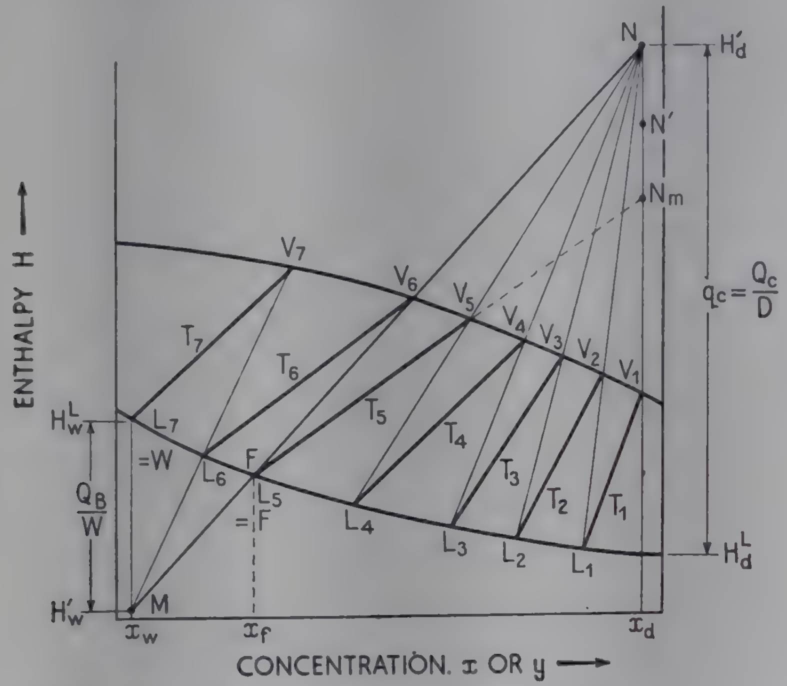


Fig. 18.47. Determination of number of plates using enthalpy-composition diagram

## Determination of the Number of Plates on *H*-*x* Diagram

The method adopted for determining the necessary number of plates for a desired separation is indicated in Fig. 18.47. The position of the feed $[F, \; x_f]$ is shown at $F$ on the boiling line and the pole $N$ is located as $[x_d, H_d']$, where $H_d' = H_d^L + \dfrac{Q_C}{D}$. Pole $M$ is located as on the extension of $NF$ cutting the ordinate at $x_w$ in $M$.

The condition of the vapour leaving the top plate is shown at $V_1$ on the dew-point curve with abscissa $x_d$. The condition of the liquid on the top plate is then found by drawing the tie line $T_1$ from $V_1$ to $L_1$ on the boiling curve. The condition $V_2$ of the vapour on second plate is found (equation 18.103) by drawing $L_1N$ to cut the dew-point curve on $V_2$. $L_2$ is then found on the tie line $T_2$. The vapour and liquid conditions $V_3$, $V_4$, $V_5$ and $L_3$, $L_4$ are found in the same way. Tie line $T_5$ gives $L_5$, which has the same composition as the feed. $V_6$ is then found using the line $MFV_6$, as this represents the vapour on the top plate of the rectifying section. $L_6$, $L_7$ and $V_7$ are then found by similar construction. $L_7$ has the required composition $x_w$ of the bottoms.

As an alternative procedure we can start with the feed condition and work up and down the column.

### Minimum Reflux Ratio

The pole $N$ has co-ordinates $\left(x_d, H_d^L + \dfrac{Q_C}{D}\right)$. Writing $q_c = \dfrac{Q_C}{D}$ as the heat removed in the condenser per unit mass of product as liquid at the boiling point, then $q_c$ is represented in Fig. 18.47 as shown. The number of plates in the rectifying section is determined, for a given feed $x_f$ and product $x_d$, by the height of this pole $N$. As $N$ is lowered to say $N'$ the heat $q_c$ falls, but the number of plates required increases. When $N$ lies at $N_m$ on the isothermal through

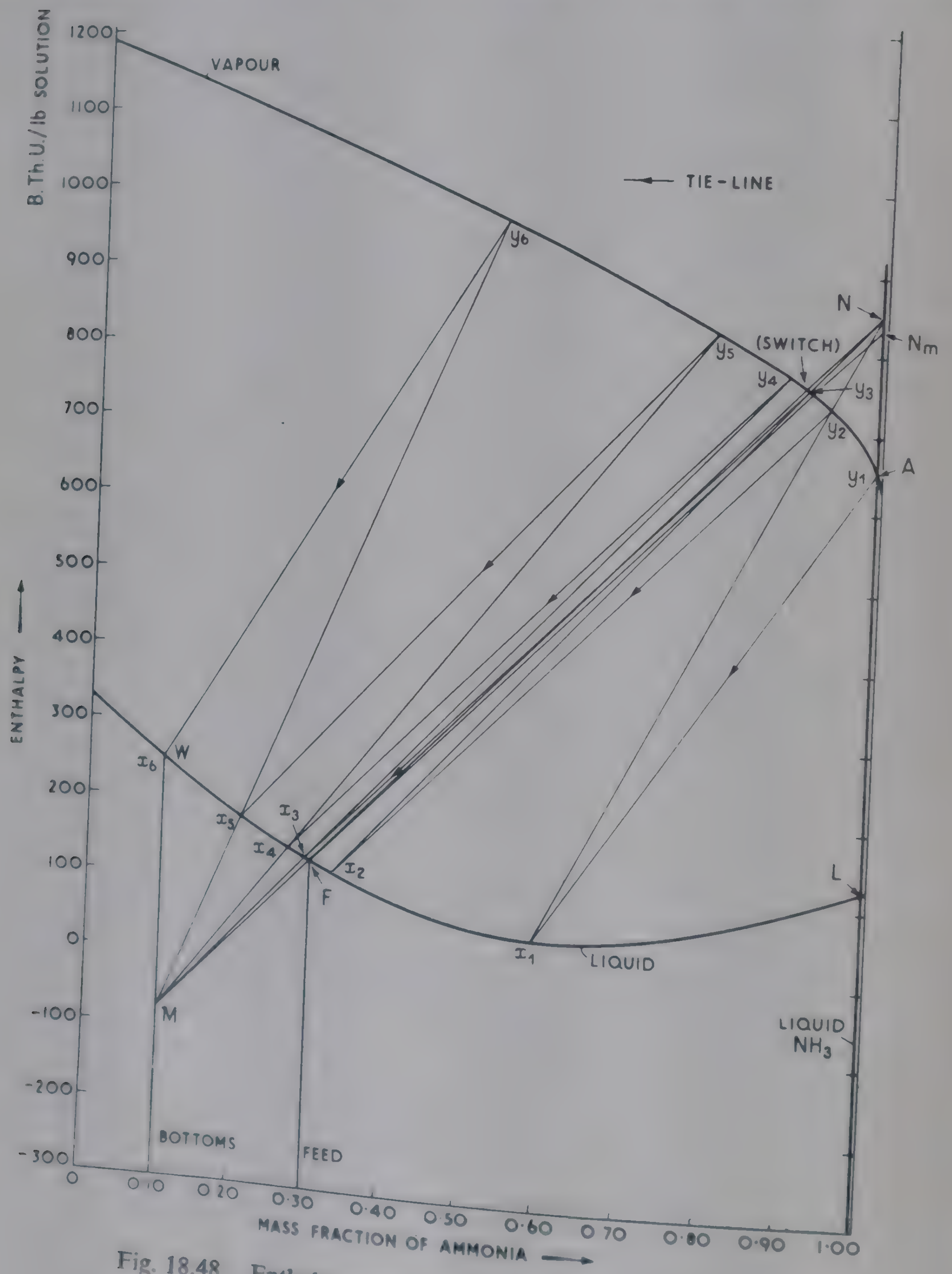


Fig. 18.48. Enthalpy-composition diagram for ammonia-water 10 atmospheres absolute pressure

$F$, $q_c$ is a minimum but the number of plates required becomes infinite. Since the tie lines have different slopes, it follows that there is a minimum reflux for each plate, and the tie line cutting the vertical axis at the highest value of $H$ will give the minimum practical reflux. This, as indicated above, will frequently correspond to the tie line through $F$.

From equations 18.109,

$$\frac{H_d{}^L + q_c - H_n{}^V}{H_n{}^V - H_{n-1}^L} = \frac{x_d - y_n}{y_n - x_{n-1}} \qquad \text{....(18.121)}$$

or

$$q_c = (H_n{}^V - H_{n-1}^L)\left(\frac{x_d - y_n}{y_n - x_{n-1}}\right) + H_n{}^V - H_d{}^L \qquad \text{....(18.122)}$$

and

$$(q_c)_{\min} = (H_f{}^V - H_{f-1}^L)\left(\frac{x_d - y_f}{y_f - x_{f-1}}\right) + H_f{}^V - H_d{}^L \qquad \text{....(18.123)}$$

The advantage of the $H$-$x$ chart lies in the fact that the heat quantities required for the distillation are clearly indicated. Thus, the higher the reflux ratio the more heat must be removed per mol of product, and point $N$ rises. This shows at once that both $q_c$ and $Q_B$ are increased. The use of this method will be illustrated by considering the separation of ammonia from an ammonia-water mixture, as occurs in the ammonia absorption unit for refrigeration.

**Example.** It is required to separate 1 ton/hour of a solution of ammonia in water, containing 30% by weight of ammonia, to give a top product of 99·5% purity and a weak solution containing 10% by weight of ammonia.

Calculate the heat required in the boiler and the heat to be rejected in the condenser, assuming a reflux 8% in excess of the minimum and a column pressure of 10 atmospheres absolute. The plates may be assumed to have an ideal efficiency of 60%.

**Solution.** Taking a material balance for the whole throughput and for the ammonia gives:

$$D + W = 2{,}240$$

$$0{\cdot}995D + 0{\cdot}1W = 2{,}240 \times 0{\cdot}3.$$

Thus $$D = 500 \text{ lb/hr}$$

and $$W = 1{,}740 \text{ lb/hr}$$

The enthalpy-composition chart for this system is shown in Fig. 18.48. It is assumed that the feed $F$ and the bottom product $W$ are liquids at their boiling points.

*Location of the poles N and M*

$N_m$ for minimum reflux will be found by drawing a tie-line through $F$, representing the feed, to cut the line $x = 0{\cdot}995$ at $N_m$.

$$\text{The minimum reflux ratio } R_m = \frac{\text{length } N_mA}{\text{length } AL}$$

$$= \frac{839 - 665}{665 - 127} = 0{\cdot}323$$

Since the actual reflux is 8% above the minimum,

$$NA = 1{\cdot}08 N_m A$$
$$= 1{\cdot}08 \times 174 = 188$$

Point $N$ therefore has an ordinate $188 + 665 = 853$ and abscissa 0·995.

Point $M$ is found by drawing $NF$ to cut the line $x = 0{\cdot}10$, through $W$, at $M$.

Then the number of theoretical plates is found, as on the diagram, to be 5+.

The number of plates to be provided $= \frac{5}{0{\cdot}6} = 8{\cdot}33$, say 9.

The feed is introduced just below the third ideal plate from the top, or just below the fifth actual plate.

Heat input at the boiler per unit mass of bottom product

$$= \frac{Q_B}{W} = 250 - (-90) = 340$$

Heat input to boiler $= 340 \times 1{,}740 = \underline{\underline{592{,}000 \text{ B.Th.U./hr.}}}$

Condenser duty $=$ length $NL \times D$

$$= (853 - 127) \times 500 = \underline{\underline{363{,}000 \text{ B.Th.U./hr.}}}$$

## BATCH DISTILLATION

In the previous sections we have considered conditions in which there has been a continuous feed to the still and a continuous withdrawal of products from the top and bottom. In many instances chemical processes are carried out in batches, and it is more convenient to distil each batch separately. In these cases the whole of a batch is run into the boiler of the still and, on heating, the vapour is passed into a fractionation column, as indicated in Fig. 18.49. As with continuous distillation, the composition of the top product will depend on the still composition, the number of plates in the column and on the reflux ratio used. When the still is operating, since the top product will be relatively rich in the more volatile component, the liquid remaining in the still will become steadily weaker in this component. As a result, the purity of the top product will steadily fall. Thus, the still may be charged with $S_1$ mols of a mixture containing a mol fraction $x_{s1}$ of the more volatile component. Initially, with a reflux ratio $R_1$, the top product has a composition $x_{d1}$. If after a certain interval of time the composition of the top product starts to fall, then, if we increase the reflux ratio to a new value $R_2$, it will be possible to obtain the same composition at the top as before, although the composition in the still is weakened to $x_{s2}$. This method of operating a batch still requires a continuous increase in the reflux ratio to maintain a constant quality of the top product.

An alternative method of procedure is to work with a constant reflux ratio and allow the composition of the top product to fall. For example, if a product of composition 0·9 with respect to the more volatile component is required, then we may arrange that the composition initially obtained is 0·95, and allow distillation to continue until the composition has fallen to 0·85. The total product obtained will then have the required composition, provided the amounts of a given purity are correctly chosen.

One of the added merits of batch distillation lies in the fact that more than one product may be obtained. Thus, we can distil a binary mixture of alcohol and water and obtain initially a high quality alcohol. As the composition in the still weakens with respect to alcohol, we can remove a second product from the top with a reduced concentration of alcohol. In this way it is possible to obtain not only two different quality products, but to reduce the alcohol in the still to a minimum value. This method of operation is particularly useful for handling small quantities of multi-component organic mixtures, since it is possible to obtain the different components at reasonable degrees of purity, one after the other. To obtain the maximum recovery of a valuable component, the charge remaining in the still after the first distillation may be added to the next batch, and re-distilled a second time.

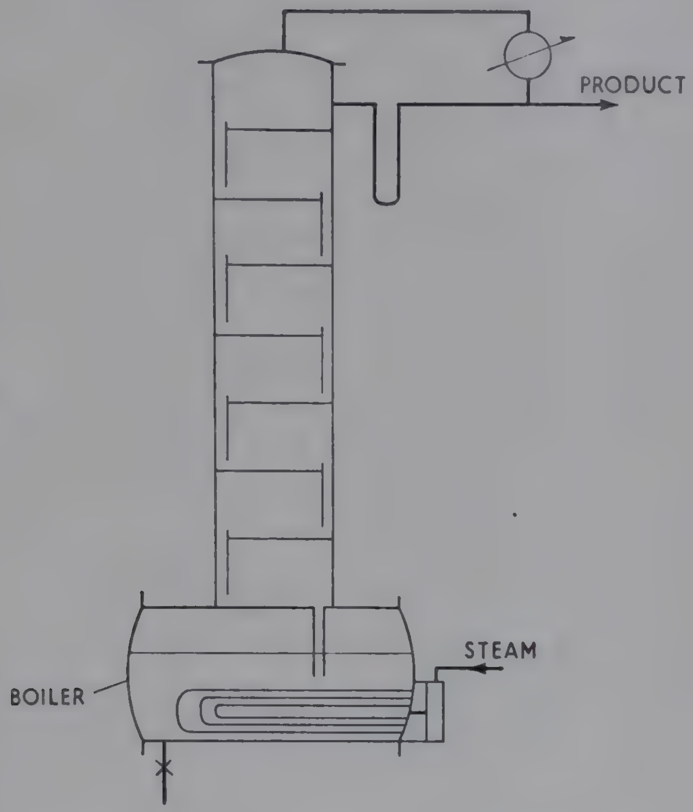


Fig. 18.49. Column for batch distillation

### Operation at Constant Product Composition

Suppose a column with four ideal plates be used to separate a mixture of ethyl alcohol and water. Initially there are in the still $S_1$ mols of liquor of mol fraction $x_{s1}$ with respect to the more volatile component (alcohol). The top product is to contain a mol fraction $x_d$, and this necessitates a reflux ratio $R_1$. Suppose the distillation to be continued till there are $S_2$ mols in the still, of mol fraction $x_{s2}$. Then, for the same number of plates, the reflux ratio will have been increased to $R_2$. If the amount of product obtained is $D_b$ mols, by a material balance we have:—

$$S_1 \, . \, x_{s1} - S_2 \, . \, x_{s2} = D_b \, . \, x_d \qquad \ldots .(18.124)$$

and

$$S_1 - S_2 = D_b \qquad \ldots .(18.125)$$

$$\therefore \quad S_1 \, . \, x_{s1} - (S_1 - D_b) \, . \, x_{s2} = D_b \, . \, x_d$$

$$\therefore \quad S_1 \, . \, x_{s1} - S_1 \, . \, x_{s2} = D_b \, . \, x_d - D_b \, . \, x_{s2}$$

$$\therefore \quad D_b = S_1 \left[ \frac{x_{s1} - x_{s2}}{x_d - x_{s2}} \right] = \frac{a}{b} \qquad \ldots .(18.126)$$

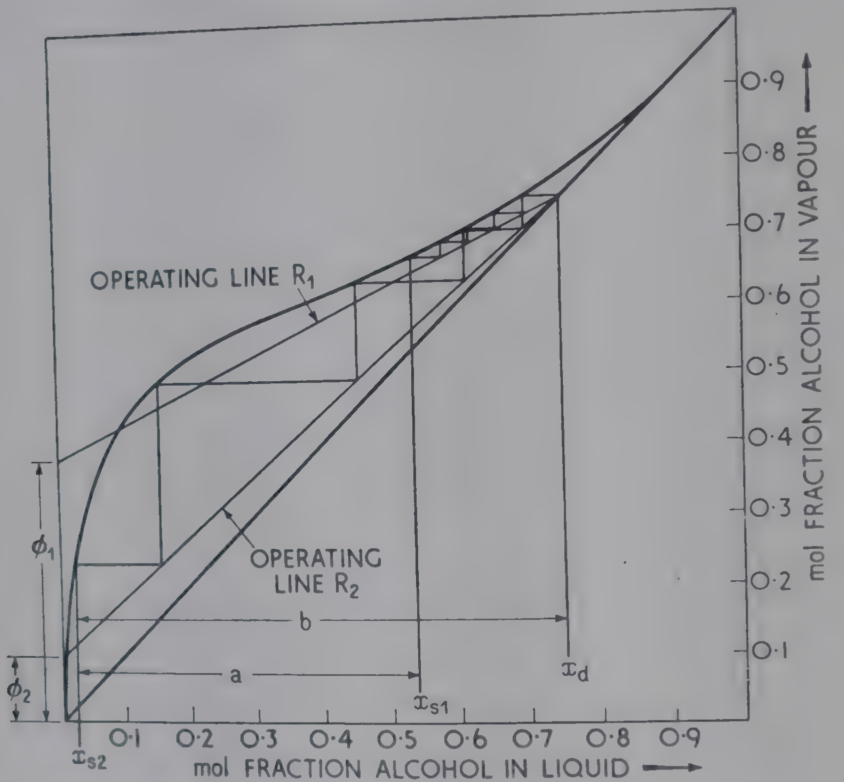


Fig. 18.50. Graphical representation of batch distillation

where *a* and *b* are as shown in Fig. 18.50. If $\phi$ is the intercept on the *Y*-axis for any operating line (equation 18.29), then

$$\frac{x_d}{R+1} = \phi \ , \quad \text{or} \quad R = \frac{x_d}{\phi} - 1 \qquad \ldots .(18.127)$$

These equations enable the final reflux ratio to be found for any desired end concentration in the still, and also give the total quantity of distillate obtained. What is important, in comparing the operation at constant reflux ratio with that at constant product composition, is the difference in the total amount of steam used in the distillation, for a given quantity $D_b$ of product.

If we assume that the reflux ratio *R* is adjusted continuously to keep the top product at constant quality, then at any moment the reflux ratio is given by

$R = \dfrac{\mathrm{d}L_b}{\mathrm{d}D_b}$. During the course of the distillation, the total reflux liquor flowing down the column is given by

$$\int_0^{L_b} \mathrm{d}L_b = \int_{R=R_1}^{R=R_2} R \, . \, \mathrm{d}D_b \qquad \ldots .(18.128)$$

To provide reflux $\mathrm{d}L_b$ requires the removal of heat $\lambda \, . \, \mathrm{d}L_b$ in the condenser, where $\lambda$ is the latent heat per mol. Thus, the heat to be supplied in the boiler $Q_R$ to provide this reflux during the total distillation is given by:

$$Q_R = \lambda \int_0^{L_b} \mathrm{d}L_b = \lambda \int_{R_1}^{R_2} R \, . \, \mathrm{d}D_b \qquad \ldots .(18.129)$$

This equation can be integrated graphically if we first find the relation between $R$ and $D_b$. For any desired value of $R$ we can obtain $x_s$ by drawing the operating line, and marking off the steps corresponding to the given number of stages. The amount of product $D_b$ is then obtained from equation 18.126 and, if the corresponding values of $R$ and $D_b$ are plotted, graphical integration will give the value of $\int R \, . \, \mathrm{d}D_b$.

We can find the minimum reflux ratio $R_m$ for any given still concentration $x_s$ from equation 18.37.

### Operation at Constant Reflux Ratio $R$

If the same column as before is operated at a constant reflux ratio $R$, the concentration of the M.V.C. in the top product will continuously fall. Over a small interval of time $\mathrm{d}t$, the top product composition will fall from $x_d$ to $x_d - \mathrm{d}x_d$. If in this time the amount of product obtained is $\mathrm{d}D_b$, a material balance on the more volatile component gives:

$$\text{More volatile component removed in product} = \mathrm{d}D_b \left[ x_d - \frac{\mathrm{d}x_d}{2} \right]$$

$$= x_d \, . \, \mathrm{d}D_b \qquad \ldots .(18.130)$$

and $\qquad x_d \, . \, \mathrm{d}D_b = -\mathrm{d}(S \, . \, x_s)$

But $\mathrm{d}D_b = -\mathrm{d}S$,

$\therefore \qquad -x_d \, . \, \mathrm{d}S = -S \, . \, \mathrm{d}x_s - x_s \, \mathrm{d}S$

$\therefore \qquad S \, . \, \mathrm{d}x_s = \mathrm{d}S(x_d - x_s)$

and
$$\int_{S_1}^{S_2} \frac{\mathrm{d}S}{S} = \int_{x_{s1}}^{x_{s2}} \frac{\mathrm{d}x_s}{x_d - x_s}$$

$$\ln \frac{S_1}{S_2} = \int_{x_{s2}}^{x_{s1}} \frac{\mathrm{d}x_s}{x_d - x_s} \qquad \ldots .(18.131)$$

The right-hand side of this equation can be integrated by plotting $\dfrac{1}{x_d - x_s}$ vs $x_s$. This will enable the ratio of the initial to final quantity in the still to be found for any desired change in $x_s$, and hence the amount of distillate $D_b$. The heat to be supplied to provide the reflux will now be $Q_R = \lambda \, . \, R \, . \, D_b$, and hence the reboil heat required per mol of product can be compared with that of the first method.

**Example.** A mixture of ethyl alcohol and water with 0·55 mol fraction of alcohol is distilled to give a top product of 0·75 mol fraction of alcohol. The column has four ideal plates and the distillation is stopped when the reflux ratio has to be increased beyond 4·0.

It is required to find the amount of distillate obtained, and the heat required per 100 mols of feed.

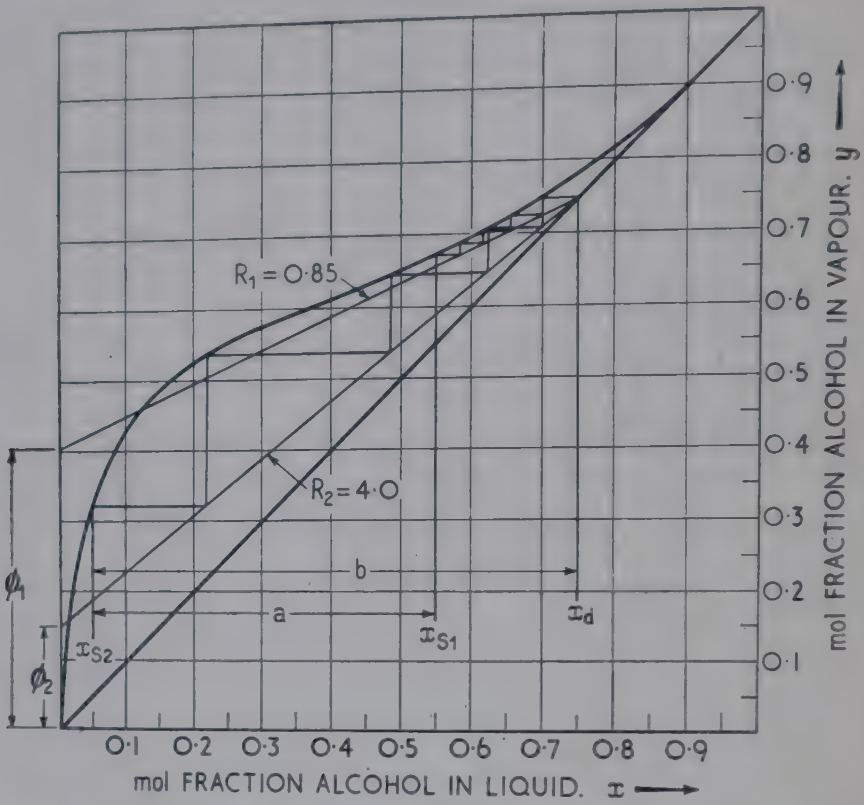


Fig. 18.51. Batch distillation—constant product composition

**Solution.** For various values of $R$ the corresponding values of the intercept $\phi$, and the concentration in the still $x_s$ are given in the table. Values of $x_s$ are found as indicated in Fig. 18.51 for the two values of $R$ of 0·85 and 4. Then the amount of product is found from equation 18.126.

Thus for $R$ of 4,

$$D_b = 100\left[\frac{0\cdot55 - 0\cdot05}{0\cdot75 - 0\cdot05}\right] = 100\left(\frac{0\cdot5}{0\cdot7}\right) = 71\cdot4 \text{ mols.}$$

Values of $D_b$ found in this way are also given in the table.

| $R$ | $\phi$ | $x_s$ | $D_b$ |
|---|---|---|---|
| 0·85 | 0·405 | 0·55 | 0 |
| 1·0 | 0·375 | 0·50 | 20 |
| 1·5 | 0·3 | 0·37 | 47·4 |
| 2·0 | 0·25 | 0·20 | 63·8 |
| 3·0 | 0·187 | 0·075 | 70·5 |
| 4 | 0·15 | 0·05 | 71·4 |

The relation between $D_b$ and $R$ is shown in Fig. 18.52 and the $\int_{R=0\cdot85}^{R=4\cdot0} R\,.\,dD_b$ is given by area $OABC$ = 96 mols.

Taking an average latent heat per pound mol for the alcohol water mixtures as 1,720 B.Th.U. the heat to be supplied to provide the reflux $Q_R$ is 96 × 1720 or approximately 165,000 B.Th.U.

The heat to be supplied to provide the reflux per mol of product is then 165,000/71·4 or 2,310 B.Th.U. and the total heat per mol of product is 2,310 + 1,720 = 4,030 B.Th.U.

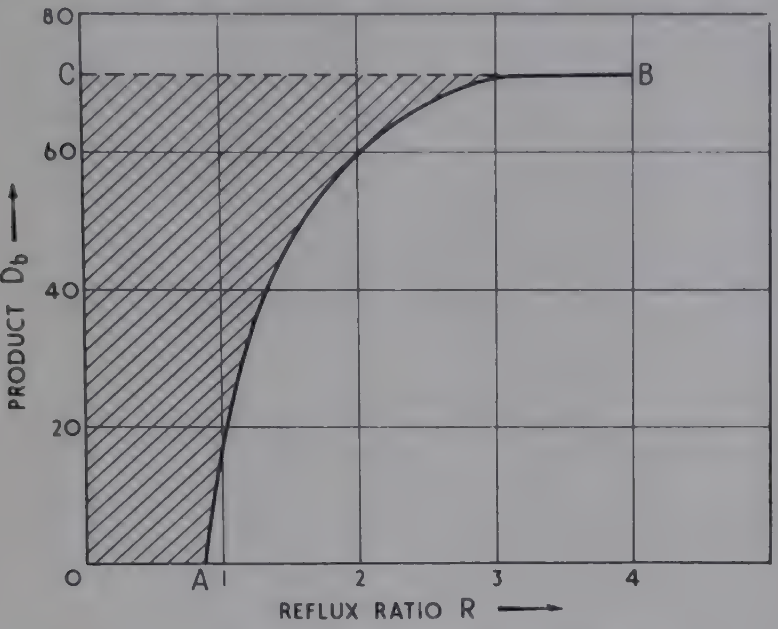


Fig. 18.52. Graphical integration

**Example 2.** If the same batch as above is distilled with a constant reflux ratio $R$ of 2·1, what will be the heat required and the average composition of the distillate if the distillation is stopped when the composition in the still has fallen to 0·105 mol fraction of alcohol?

**Solution.** The initial composition of the top product will be 0·78 as shown in Fig. 18.53 and

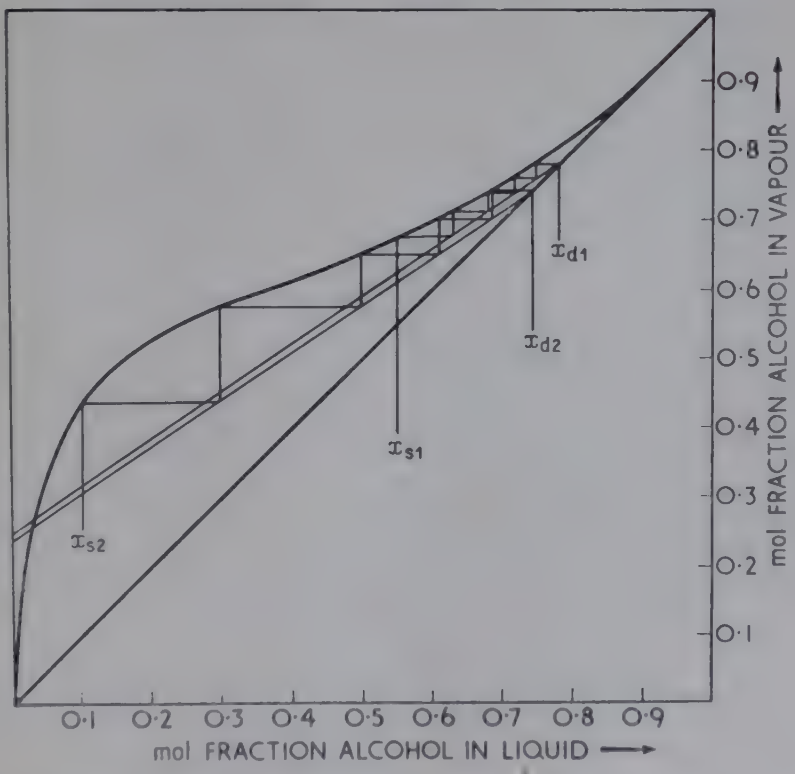

$x_{d1}$
$x_{d2}$
$x_{s1}$
$x_{s2}$
0·1
0·2
0·3
0·4
0·5
0·6
0·7
0·8
0·9
mol FRACTION ALCOHOL IN LIQUID →
0·9
0·8
0·7
0·6
0·5
0·4
0·3
0·2
0·1
mol FRACTION ALCOHOL IN VAPOUR →

Fig. 18.53. Batch distillation—constant reflux ratio

the final composition will be 0·74. Values of $x_d$, $x_s$, $x_d - x_s$ and of $\frac{1}{x_d - x_s}$ for various values of $x_s$ and a constant reflux ratio are given in the table.

*Ethyl Alcohol-Water distillation at constant R of 2·1 with column of 4 ideal plates*

| *Values of* $x_s$ | $x_d$ | $x_d - x_s$ | $\frac{1}{x_d - x_s}$ |
|---|---|---|---|
| 0·55 | 0·78 | 0·23 | 4·35 |
| 0·50 | 0·775 | 0·275 | 3·65 |
| 0·425 | 0·770 | 0·345 | 2·90 |
| 0·310 | 0·760 | 0·450 | 2·22 |
| 0·225 | 0·750 | 0·525 | 1·91 |
| 0·105 | 0·740 | 0·635 | 1·58 |

Values of $x_s$ and $\frac{1}{x_d - x_s}$ are plotted in Fig. 18.54 from which $\int_{\cdot 105}^{\cdot 55} \frac{dx_s}{x_d - x_s}$ is found to be 1·1

From equation 18.131, $\ln \frac{S_1}{S_2} = 1{\cdot}1$ and $\frac{S_1}{S_2} = 3{\cdot}0$.

product obtained $D_b = S_1 - S_2 = 100 - \frac{100}{3} = 66\frac{2}{3}$ lb. mols.

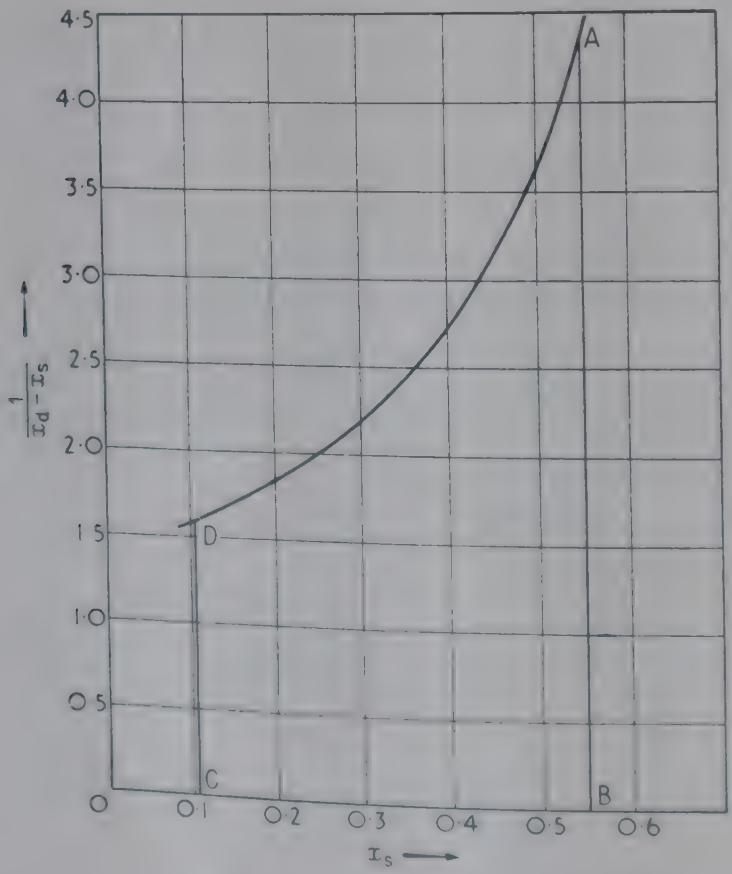


Fig. 18.54. Graphical integration

$$\text{Amount of alcohol in product} = x_1 \,.\, S_1 - x_2 \,.\, S_2$$
$$= 0{\cdot}55 \times 100 - 0{\cdot}105 \times 33{\cdot}3$$
$$= 55 - 3{\cdot}5 = 51{\cdot}5 \text{ mols}$$

$\therefore$ Average composition of product is $\dfrac{51{\cdot}5}{66{\cdot}7} = \underline{\underline{0{\cdot}77 \text{ mol fraction alcohol.}}}$

The heat required to provide the reflux is $1720 \times 2{\cdot}1 \times 66\tfrac{2}{3} = 240{,}800$ B.Th.U.

Heat required to provide reflux per mol of product $= \dfrac{240{,}800}{66{\cdot}67} = \underline{\underline{3{,}600 \text{ B.Th.U.}}}$

Thus in example 1 the total heat required per mol of product is 2,310 + 1,720 = 4,030 B.Th.U. and at constant reflux rate it is 3,600 + 1,720 = 5,320 B.Th.U., but the average quality of product is 0·77 for the second case and only 0·75 for the first.

A discussion on the relative merits of batch and continuous distillation is given by Ellis[47]. He shows that when a large number of plates are used and the reflux ratio approaches the minimum value, then continuous distillation has the lowest reflux requirement and hence operating costs. If a smaller number of plates are used and a high purity product is not desired, then the advantages will probably be with batch distillation.

## MULTICOMPONENT MIXTURES

For a binary mixture under constant pressure conditions the vapour-liquid equilibrium curve for either component is unique so that, if the concentration of either component is known in the liquid phase, the compositions of the liquid and of the vapour are fixed. It is on the basis of this single equilibrium curve that the McCabe-Thiele method was developed for the rapid determination of the number of theoretical plates required for a given separation. With a ternary system the conditions of equilibrium are more complex, for at constant pressure the mol fraction of two of the components in the liquid phase must be given before the composition of the vapour in equilibrium can be determined, even for an ideal system. Thus, the mol fraction $y_A$ in the vapour depends not only on $x_A$ in the liquid, but also on the relative properties of the other two components.

To obtain experimentally the equilibrium relationships for a multicomponent mixture requires considerable data, and one of two methods of simplification are usually adopted. For many systems, particularly those consisting of chemically similar substances, the relative volatilities of the components remain constant over a wide range of temperature and composition. This is illustrated in the table below for mixtures of phenol, ortho and meta-cresols, and xylenols, where the volatilities are shown relative to ortho-cresol.

Table 18.4. *Volatilities relative to o-cresol*

| | *Temperatures °C* | | |
|---|---|---|---|
| | 80 | 120 | 160 |
| Phenol | 1·25 | 1·25 | 1·25 |
| *o*-Cresol | 1 | 1 | 1 |
| *m*-Cresol | 0·57 | 0·62 | 0·7 |
| xylenols | 0·3 | 0·38 | 0·42 |

An alternative method, particularly useful for multicomponent mixtures of hydrocarbons in the petroleum industry, is to use the simple relation $y_A = Kx_A$. These $K$ values have been measured for a wide range of hydrocarbons at various pressures, and some values are shown in Fig. 18.55.

Some progress has been made in presenting methods for calculating ternary data from known data for the binary mixtures, but as yet no entirely satisfactory method is available.

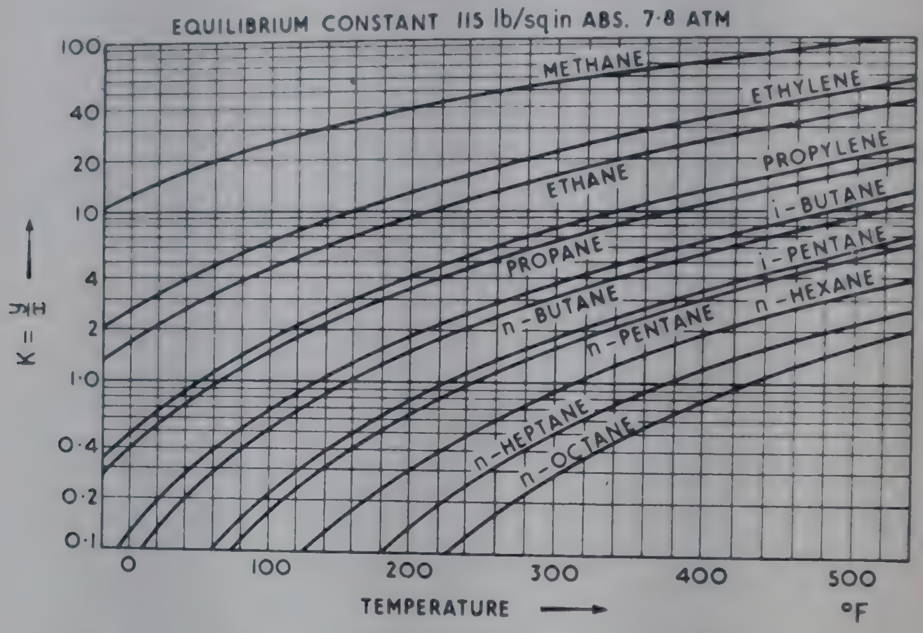


Fig. 18.55. Vapour-liquid equilibrium data for hydrocarbons

### The Feed and Product Compositions

With a binary system, if the feed composition $x_f$ and the top product composition $x_d$ are known for one component, then the composition of the bottoms $x_w$ can have any desired value, and a material balance will determine the amounts of the top and bottom products $D$ and $W$. This freedom of selecting the compositions does not apply for mixtures with three or more components. GILLILAND and REED(29) have determined the number of degrees of freedom for the continuous distillation of a multicomponent mixture. For the common case in which the feed composition, nature of the feed, and operating pressure are given, there remain only four variables that may be selected. If the reflux ratio $R$ is fixed and the number of plates above and below the feed plate chosen to give the best use of the plates, then only two variables remain. The complete composition of neither the top nor bottom product can then be fixed at will. This means that some degree of trial and error is unavoidable in calculating the number of plates required for any desired separation. Thus, if a trial composition is taken, and it is found that for the given bottom composition the desired top composition is not obtained with the selected reflux ratio, then an adjustment must be made in the bottom composition. An exact fit in a calculation of this kind is not essential since the equilibrium data and the plate efficiency will be known with only limited accuracy. This problem is frequently simplified if a sharp cut is to be made between the components, so that all of the more volatile components appear in the top and all of the less volatile in the bottom product.

### Light and Heavy Key Components

In the fractionation of multicomponent mixtures it frequently happens that the essential requirement is the separation between two components. Such components are called the key components and by concentrating attention on these it is possible to simplify the handling of complex mixtures. Suppose a four component mixture A-B-C-D, in which $A$ is the most volatile and $D$ the least volatile, to be separated as shown in the table below:

| *Feed* | *Top product* | *Bottoms* |
|---|---|---|
| $A$ | $A$ | |
| $B$ | $B$ | $B$ |
| $C$ | $C$ | $C$ |
| $D$ | | $D$ |

Then $B$ is the lightest component appearing in the bottoms and is termed the light key component, and $C$ is the heaviest component appearing in the distillate and is called the heavy key component. The main purpose of the fractionation is the separation of $B$ from $C$.

### The Calculation of the Number of Plates Required for a Given Separation

One of the most successful methods for calculating the number of plates necessary for a given separation is due to LEWIS and co-workers(12). This is based on the Sorel-Lewis method, previously described for binary mixtures. If the composition of the liquid on any plate is known, then the composition of the vapour in equilibrium is calculated from a knowledge of the vapour pressures or relative volatilities of the individual components. The composition of the liquid on the plate above is then found by using an operating equation, as for binary mixtures, but in this case there will be a separate equation for each component.

Suppose a mixture of components $A$, $B$, $C$, $D$, etc., to have mol fraction $x_A$, $x_B$, $x_C$, $x_D$, etc., in the liquid and $y_A$, $y_B$, $y_C$, $y_D$, etc., in the vapour.

Then
$$y_A + y_B + y_C + y_D + \ldots = 1 \qquad \ldots.(18.132)$$

and
$$\frac{y_A}{y_B} + \frac{y_B}{y_B} + \frac{y_C}{y_B} + \frac{y_D}{y_B} + \ldots = \frac{1}{y_B}$$

$$\therefore \quad \alpha_{AB} \cdot \frac{x_A}{x_B} + \alpha_{BB} \cdot \frac{x_B}{x_B} + \alpha_{CB} \cdot \frac{x_C}{x_B} + \alpha_{DB} \cdot \frac{x_D}{x_B} + \ldots = \frac{1}{y_B}$$

$$\therefore \qquad \sum\alpha_{AB}\cdot x_A = \frac{x_B}{y_B}$$

$$\left.\begin{aligned} y_B &= \frac{x_B}{\sum\alpha_{AB}\cdot x_A} \\ \text{And, similarly,} \qquad y_A &= \frac{x_A\cdot\alpha_{AB}}{\sum\alpha_{AB}x_A}; \qquad y_C = \frac{x_C\cdot\alpha_{CB}}{\sum\alpha_{AB}\cdot x_A} \end{aligned}\right\} \quad \ldots.(18.133)$$

Thus, the composition of the vapour is conveniently found from that of the liquid by use of the relative volatilities of the components.

**Example.** A mixture of ortho, meta, and para mononitrotoluenes containing 60, 4, and 36 mol % respectively of the three isomers is to be continuously distilled to give a top product of 98 mol % ortho, and the bottom is to contain 12·5 mol % ortho. The mixture is to be distilled at a temperature of 137°C requiring a pressure in the boiler of about 45 mm of mercury. If a reflux ratio of 5 is used, how many plates will be required and what will be the approximate compositions of the product streams?

**Solution.** The volatility of ortho relative to the para isomer can be taken as 1·70 and of the meta as 1·16 over the temperature range of 110° to 140°C. As a first estimate, suppose the distillate to contain 0·6 mol % meta and 1·4 mol % para. Then a material balance will give the composition of the bottoms.

Consider 100 mols of feed.

Let $D$ and $W$ be the mols of product and bottoms, and $x_{do}$ and $x_{wo}$ the mol fraction of the ortho in the distillate and bottoms.

Total material balance gives:— $100 = D + W$

Ortho balance gives:— $60 = D\,.\,x_{do} + W\,.\,x_{wo}$

whence $60 = (100 - W)0{\cdot}98 + W\,0{\cdot}125$

from which $D = 55{\cdot}56$ and $W = 44{\cdot}44$.

The material balance to give the composition and amount of the streams can then be obtained as,

| *Component* | *Feed* | | *Distillate* | | *Bottoms* | |
|---|---|---|---|---|---|---|
| | *Mols* | *Mol %* | *Mols* | *Mol %* | *Mols* | *Mol %* |
| Ortho $o$ | 60 | 60 | 54·44 | 98·0 | 5·56 | 12·5 |
| Meta $m$ | 4 | 4 | 0·33 | 0·6 | 3·67 | 8·3 |
| Para $p$ | 36 | 36 | 0·79 | 1·4 | 35·21 | 79·2 |
| | 100 | 100 | 55·56 | 100 | 44·44 | 100 |

*Equations of operating lines.* The liquid and vapour streams in the column can be obtained as,

Above feed-point:

Liquid Downflow $L_n = 5\,.\,D = 277{\cdot}8$

Vapour Up $V_n = 6\,.\,D = 333{\cdot}4$

Below feed-point, taking all the feed as liquid at its boiling point:

Liquid Downflow $L_m = L_n + F = 277{\cdot}8 + 100 = 377{\cdot}8$

Vapour Up $V_m = L_m - W = 377{\cdot}8 - 44{\cdot}44 = 333{\cdot}4$

The equations for the operating lines below the feed plate can then be written as,

$$\left.\begin{aligned}
& y_{mo} = \frac{L_m}{V_m} \,.\, x_{m+1} - \frac{W}{V_m} \,.\, x_w \\
\text{Ortho} \qquad & y_{mo} = \frac{377{\cdot}8}{333{\cdot}4} \,.\, x_{m+1} - \frac{44{\cdot}44}{333{\cdot}4} \,.\, x_w \\
& \phantom{y_{mo}} = 1{\cdot}133\, x_{m+1} - 0{\cdot}0166 \\
\text{Meta} \qquad & y_{mm} = 1{\cdot}133\, x_{m+1} - 0{\cdot}011 \\
\text{Para} \qquad & y_{mp} = 1{\cdot}133\, x_{m+1} - 0{\cdot}105
\end{aligned}\right\} \qquad \ldots.(18.134)$$

And above the Feed Plate:—

$$\left.\begin{aligned}
& y_n = \frac{L_n}{V_n} \,.\, x_{n+1} + \frac{D}{V_n} \,.\, x_d \\
\text{Ortho} \qquad & y_{no} = \frac{277{\cdot}8}{333{\cdot}4} \,.\, x_n + \frac{55{\cdot}56}{333{\cdot}4} \times 0{\cdot}98 \\
& \phantom{y_{no}} = 0{\cdot}833\, x_n + 0{\cdot}0163 \\
\text{Meta} \qquad & y_{nm} = 0{\cdot}833\, x_n + 0{\cdot}001 \\
\text{Para} \qquad & y_{np} = 0{\cdot}833\, x_n + 0{\cdot}002
\end{aligned}\right\} \qquad \ldots.(18.135)$$

*Composition of Liquid on First Plate.* The temperature of distillation has been fixed by considerations of safety at 137°C and, from a knowledge of the vapour pressures of the three components, the pressure in the still is found to be about 45 mm of Mercury. The composition of the vapour in the still is found from the relation $y_{so} = \dfrac{\alpha_o \,.\, x_{so}}{\Sigma \alpha x_s}$.

The liquid composition on the first plate is then found from equation 18.134; thus for ortho:

$$0{\cdot}191 = 1{\cdot}133\, x_1 - 0{\cdot}0166$$

$$x_1 = 0{\cdot}183$$

The values of the plate compositions as found in this way are shown in the table below. The liquid on plate 7 has a composition with the ratio of the concentrations of ortho and para about that in the feed, and the feed will therefore be introduced on this plate. Above this plate the same method is used but the operating equations are those of equations 18.135. The vapour from the sixteenth plate has the required concentration of the ortho isomer, and the values for the meta and para are sufficiently near to take this as showing that sixteen ideal plates will be required.

Using relation $(y_m)_o = \dfrac{\alpha_o \,.\, x_{mo}}{\Sigma \alpha x_m}$

*Plate compositions below the feed plate*

| *Component* | $x_s$ | $\alpha x_s$ | $y_s$ | $x_1$ | $\alpha x_1$ | $y_1$ | $x_2$ |
|---|---|---|---|---|---|---|---|
| *o* | 0·125 | 0·211 | 0·191 | 0·183 | 0·308 | 0·270 | 0·253 |
| *m* | 0·083 | 0·096 | 0·088 | 0·088 | 0·102 | 0·090 | 0·089 |
| *p* | 0·792 | 0·792 | 0·721 | 0·729 | 0·729 | 0·640 | 0·658 |
| | 1 | 1·099 | 1 | 1 | 1·139 | 1 | 1 |

| | $\alpha x_2$ | $y_2$ | $x_3$ | $\alpha x_3$ | $y_3$ | $x_4$ | $\alpha x_4$ |
|---|---|---|---|---|---|---|---|
| *o* | 0·430 | 0·357 | 0·330 | 0·561 | 0·450 | 0·411 | 0·698 |
| *m* | 0·103 | 0·086 | 0·086 | 0·1 | 0·08 | 0·080 | 0·093 |
| *p* | 0·658 | 0·557 | 0·584 | 0·584 | 0·420 | 0·509 | 0·509 |
| | 1·191 | 1 | 1 | 1·245 | 1 | 1 | 1·300 |

*Plate composition below the feed plate (contd.)*

| Component | $y_4$ | $x_5$ | $\alpha x_5$ | $y_5$ | $x_6$ | $\alpha x_6$ | $y_6$ |
|---|---|---|---|---|---|---|---|
| *o* | 0·539 | 0·488 | 0·830 | 0·613 | 0·556 | 0·944 | 0·674 |
| *m* | 0·071 | 0·072 | 0·083 | 0·061 | 0·063 | 0·073 | 0·052 |
| *p* | 0·392 | 0·440 | 0·440 | 0·326 | 0·381 | 0·381 | 0·274 |
| | 1 | 1 | 1·353 | 1 | 1 | 1·398 | 1 |

| | $x_7$ |
|---|---|
| *o* | 0·609 |
| *m* | 0·055 |
| *p* | 0·336 |
| | 1 |

*Plate compositions above the feed plate*

| Component | $x_7$ | $\alpha x_7$ | $y_7$ | $x_8$ | $\alpha x_8$ | $y_8$ | $x_9$ |
|---|---|---|---|---|---|---|---|
| *o* | 0·609 | 1·035 | 0·721 | 0·669 | 1·136 | 0·770 | 0·728 |
| *m* | 0·055 | 0·064 | 0·044 | 0·051 | 0·059 | 0·040 | 0·047 |
| *p* | 0·336 | 0·336 | 0·235 | 0·280 | 0·280 | 0·190 | 0·225 |
| | 1 | 1·435 | 1 | 1 | 1·475 | 1 | 1 |

| | $\alpha x_9$ | $y_9$ | $x_{10}$ | $\alpha x_{10}$ | $y_{10}$ | $x_{11}$ | $\alpha x_{11}$ |
|---|---|---|---|---|---|---|---|
| *o* | 1·238 | 0·816 | 0·782 | 1·330 | 0·856 | 0·832 | 1·415 |
| *m* | 0·054 | 0·035 | 0·041 | 0·047 | 0·030 | 0·035 | 0·040 |
| *p* | 0·225 | 0·149 | 0·177 | 0·177 | 0·114 | 0·133 | 0·133 |
| | 1·517 | 1 | 1 | 1·554 | 1 | 1 | 1·588 |

| | $y_{11}$ | $x_{12}$ | $\alpha x_{12}$ | $y_{12}$ | $x_{13}$ | $\alpha x_{13}$ | $y_{13}$ |
|---|---|---|---|---|---|---|---|
| *o* | 0·891 | 0·874 | 1·485 | 0·920 | 0·907 | 1·542 | 0·940 |
| *m* | 0·025 | 0·029 | 0·033 | 0·020 | 0·023 | 0·027 | 0·017 |
| *p* | 0·084 | 0·097 | 0·097 | 0·060 | 0·070 | 0·070 | 0·043 |
| | 1 | 1 | 1·615 | 1 | 1 | 1·639 | 1 |

| | $x_{14}$ | $\alpha x_{14}$ | $y_{14}$ | $x_{15}$ | $\alpha x_{15}$ | $y_{15}$ | $x_{16}$ |
|---|---|---|---|---|---|---|---|
| *o* | 0·932 | 1·585 | 0·957 | 0·953 | 1·620 | 0·970 | 0·968 |
| *m* | 0·019 | 0·022 | 0·013 | 0·014 | 0·016 | 0·010 | 0·010 |
| *p* | 0·049 | 0·049 | 0·030 | 0·033 | 0·033 | 0·020 | 0·022 |
| | 1 | 1·656 | 1 | 1 | 1·669 | 1 | 1 |

| | $\alpha x_{16}$ | $y_{16}$ |
|---|---|---|
| *o* | 1·632 | 0·980 |
| *m* | 0·012 | 0·007 |
| *p* | 0·022 | 0·003 |
| | 1·666 | 1 |

## Minimum Reflux Ratio

In the distillation of binary mixtures, the minimum reflux ratio is given by the operating line which joins the product composition to the point where the *q* line cuts the equilibrium curve. Thus, if Fig. 18.56 represents the McCabe-Thiele diagram for a binary mixture of the two key components of a multicomponent mixture, then *DF* and *WF* give the minimum reflux ratios for the rectifying and stripping sections respectively. As we move along these operating lines, the change in composition on adjacent plates becomes less and less until it becomes negligible at the feed plate. At *F* the compositions are said to be "pinched." In a multicomponent mixture the presence of the components lighter than the light key will shift the operating line *DF* to some value *DN*, and the components heavier than the heavy key will shift the lower line *WN* to some line *WM*. In multicomponent distillation we have two pinched-in regions. In locating these pinched-in regions we may note that:

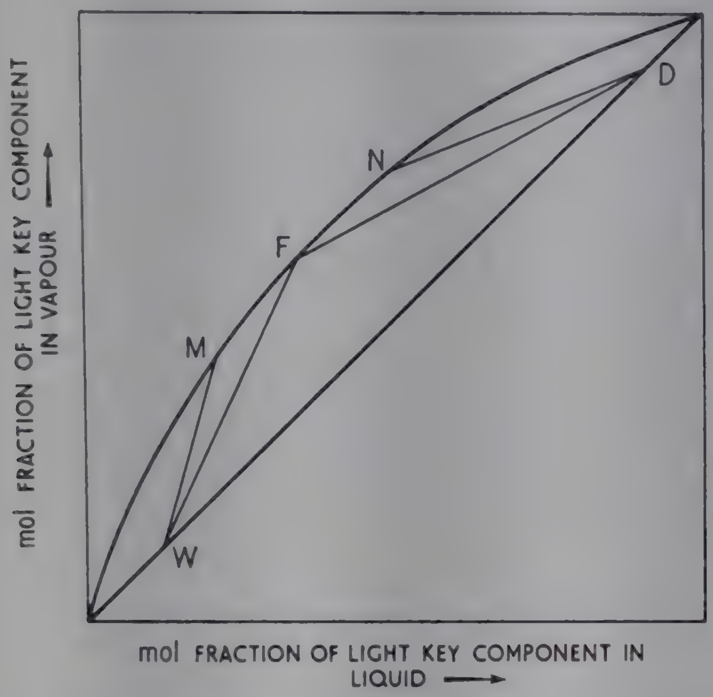

D
N
F
M
W
mol FRACTION OF LIGHT KEY COMPONENT IN VAPOUR ⟶
mol FRACTION OF LIGHT KEY COMPONENT IN LIQUID ⟶

Fig. 18.56. McCabe-Thiele diagram for two key components

*a.* If there are no components lighter than the light key, then all of the components appear in the bottoms and the pinch in the stripping section will be near the feed plate.

*b.* If there are no components heavier than the heavy key, then all of the components will appear in the top and the upper pinch is also at the feed plate.

If both of these conditions are true, then the two pinches coincide at the feed plate, as for a binary system. For the general case, a number of proposals have been made for locating the pinched regions and hence the minimum reflux ratio $R_m$. Of these, the methods of Colburn and Underwood will be mentioned. We may note that between the feed plate and the enriching pinch the concentrations of components heavier than the heavy key fall off rapidly, so that the upper pinch can be regarded as containing the heaviest key and lighter components. Similarly the lower pinch has the lightest key and heavier components.

***Colburn's Method*[25] *for Minimum Reflux $R_m$*.** Let $A$ and $B$ be the light and heavy key components of a multicomponent mixture. Then we can apply the method given earlier (equation 18.37) for binary mixtures and obtain the minimum reflux ratio $R_m$ from the relationship

$$R_m = \frac{1}{\alpha_{AB} - 1}\left\{\frac{x_{dA}}{x_{nA}} - \alpha_{AB} \cdot \frac{x_{dB}}{x_{nB}}\right\} \qquad \ldots.(18.136)$$

where $x_{dA}$ and $x_{nA}$ are the top and pinch compositions of the light key component,

$x_{dB}$ and $x_{nB}$ are the top and pinch compositions of the heavy key component, and

$\alpha_{AB}$ is the volatility of the light key relative to the heavy key component.

The difficulty in using this equation is that we only know the values of $x_{nA}$ and $x_{nB}$ in special cases where the pinch coincides with the feed composition. COLBURN has suggested that an approximate value for $x_{nA}$ is given by

$$x_{nA}\,(\text{approx}) = \frac{r_f}{(1 + r_f)(1 + \sum \alpha \,.\, x_{fh})} \qquad \ldots.(18.137)$$

and

$$x_{nB}\,(\text{approx}) = \frac{x_{nA}}{r_f} \qquad \ldots.(18.137a)$$

where $r_f$ is the estimated ratio of the key components on the feed plate. For an all liquid feed at its boiling point, $r_f$ equals the ratio of the key components in the feed. Otherwise $r_f$ is the ratio of the key components in the liquid part of the feed,

$x_{fh}$ is the mol fraction of each component in the liquid portion of feed heavier than the heavy key, and

$\alpha$ is the volatility of the component relative to the heavy key.

Using this approximate value for $R_m$, equation 18.136 can be rearranged to give the concentrations of all the light components in the upper pinch as

$$x_n = \frac{x_d}{(\alpha - 1)R_m + \alpha \,.\, \dfrac{x_{dB}}{x_{nB}}} \qquad \ldots.(18.138)$$

The concentration of the heavy key in the upper pinch is then obtained by difference, after obtaining the values for all the light components. The second term in the denominator is usually negligible, as the concentration of the heavy key in the top product is small.

A similar condition occurs in the stripping section, and the concentration of all components heavier than the light key is given by

$$x_m = \frac{\alpha_{AB} x_w}{(\alpha_{AB} - \alpha)\dfrac{L_m}{W} + \alpha \cdot \dfrac{x_{wA}}{x_{mA}}} \qquad \ldots .(18.139)$$

where $x_m$ and $x_w$ are the compositions of a given heavy component at the pinch and in the bottoms,

$x_{mA}$ and $x_{wA}$ are the compositions of the light key component at the pinch and in the bottoms,

$L_m/W$ is the molar ratio of the liquid in the stripping section to the bottom product,

$\alpha_{AB}$ is the volatility of the light key relative to the heavy key, and

$\alpha$ is the volatility of the component relative to the heavy key.

Again, the second term in the denominator may usually be neglected.

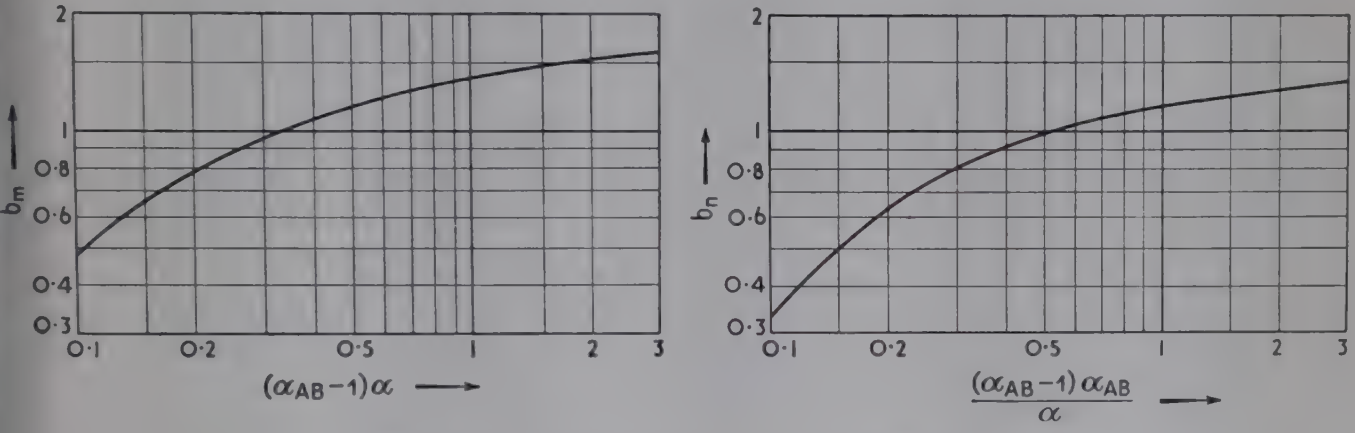


Fig. 18.57. Factors in **Colburn**'s solution

The essence of **Colburn**'s method is that he has provided an empirical relation between the compositions at the two pinches for the condition of minimum reflux. This enables the assumed value of $R_m$ to be checked. This equality may be written as

$$\frac{r_m}{r_n} = \psi = \frac{1}{(1 - \sum b_m \alpha x_m)(1 - \sum b_n x_n)} \qquad \ldots .(18.140)$$

where $r_m$ is the ratio of the light key to the heavy key in the stripping pinch,

$r_n$ is the ratio of the light key to the heavy key in the upper pinch,

$\sum b_m \alpha x_m$ is the summation of $b_m \alpha x_m$ for all components heavier than the heavy key in the lower pinch,

$\sum b_n x_n$ is the summation of $b_n x_n$ for all components lighter than the light key in upper pinch, and

$b_m$, $b_n$ are the factors shown in Fig. 18.57.

**Example (Determination of minimum reflux ratio).** A mixture of $n$-$C_4$ to $n$-$C_7$ hydrocarbons is to be distilled to give top and bottom products as shown in the table below. The distillation is effected at 115 lb/in.$^2$ abs. and the feed is at 210°F. The equilibrium values ($K$) are shown in Fig. 18.55. It is required to find the minimum reflux ratio. No cooling occurs in the condenser.

| *Component* | *Feed* | *Distillate* | | *Bottoms* | |
|---|---|---|---|---|---|
| | $F$ | $D$ | $x_d$ | $W$ | $x_w$ |
| $n$-$C_4$ (light key) | 40 | 39 | 0·975 | 1 | 0·017 |
| $C_5$ (heavy key) | 23 | 1 | 0·025 | 22 | 0·367 |
| $C_6$ | 17 | | | 17 | 0·283 |
| $C_7$ | 20 | | | 20 | 0·333 |
| | $F=100$ | $D=40$ | 1 | $W=60$ | 1 |

**Solution.**

1. *Estimation of Top Temperature $T_d$.* By dew point calculation $\Sigma x_d = \Sigma \frac{x_d}{K}$

| *Component* | $x_d$ | $T_d = 160°F$ | | $T_d = 159°F$ | |
|---|---|---|---|---|---|
| | | $K$ | $\frac{x_d}{K}$ | $K$ | $\frac{x_d}{K}$ |
| $C_4$ | 0·975 | 1·05 | 0·929 | 1·04 | 0·938 |
| $C_5$ | 0·025 | 0·41 | 0·061 | 0·405 | 0·062 |
| | | | 0·990 | | 1·000 |

Hence top temperature $T_d = 159°F$.

2. *Estimation of Still Temperature $T_s$.* $\Sigma x_w = K \, . \, x_w$.

| *Component* | $x_w$ | $T_w = 295°F$ | | $T_w = 289°F$ | |
|---|---|---|---|---|---|
| | | $K$ | $K \, . \, x_w$ | $K$ | $K \, . \, x_w$ |
| $n$-$C_4$ | 0·017 | 3·05 | 0·052 | 2·93 | 0·050 |
| $C_5$ | 0·367 | 1·6 | 0·586 | 1·54 | 0·565 |
| $C_6$ | 0·283 | 0·87 | 0·246 | 0·82 | 0·232 |
| $C_7$ | 0·333 | 0·49 | 0·163 | 0·46 | 0·153 |
| | 1 | | 1·047 | | 1·000 |

Hence the still temperature is $T_s = 289°F$.

3. *Calculation of Feed Condition.* To determine the nature of the feed we must find its boiling point $T_B$, i.e. where $\Sigma K \,.\, x_f = 1$.

| Component | $x_f$ | $T_B = 220°F$ $K$ | $Kx_f$ | $T_B = 218°F$ $K$ | $Kx_f$ |
|---|---|---|---|---|---|
| $n$-$C_4$ | 0·40 | 1·80 | 0·72 | 1·78 | 0·712 |
| $C_5$ | 0·23 | 0·81 | 0·186 | 0·79 | 0·182 |
| $C_6$ | 0·17 | 0·39 | 0·066 | 0·38 | 0·065 |
| $C_7$ | 0·20 | 0·19 | 0·0038 | 0·185 | 0·067 |
| | 1 | | 1·01 | | 0·996 |

Hence $T_B$ is approximately 218°F, and since the feed is at 210°F we may take it as all liquid at its boiling point.

4. *Calculation of Pinch Temperatures.* The temperature of the pinches are taken in the first place at one-third and two-thirds of the difference between the still and top temperatures.

Thus the upper pinch temperature $T_N = 159 + \frac{1}{3}[289 - 159] = 202°F$

and the lower pinch temperature $T_M = 159 + \frac{2}{3}[289 - 159] = 246°F$.

5. *Calculation of approximate minimum Reflux Ratio.* The calculations can be set down in the following way

| Component | $T_n = 202°F$ $\alpha$ | $T_m = 246°F$ $\alpha$ | $x_{fh}$ | $\alpha \,.\, x_{fh}$ |
|---|---|---|---|---|
| $C_4$ | 2·38 | 2·0 | | |
| $C_5$ | 1·00 | 1 | | |
| $C_6$ | 0·455 | 0·464 | 0·17 | 0·077 |
| $C_7$ | 0·22 | 0·254 | 0·20 | 0·044 |
| | | | | 0·121 |

Then

$$r_f = \frac{x_{f4}}{x_{f5}} = \frac{0{\cdot}40}{0{\cdot}23} = 1{\cdot}740.$$

From equation 18.137,

$$x_{n4} = \frac{1{\cdot}740}{(1 + 1{\cdot}740)(1 + 0{\cdot}121)} = 0{\cdot}565$$

and

$$x_{n5} = \frac{0{\cdot}565}{1{\cdot}740} = 0{\cdot}325$$

$$R_m = \frac{1}{(2{\cdot}38 - 1)}\,\frac{0{\cdot}975}{0{\cdot}563} - 2{\cdot}38\left(\frac{0{\cdot}025}{0{\cdot}325}\right)$$
$$= 1{\cdot}12.$$

6. *The Streams in the Column.*

$$L_n = D \,.\, R_m = 40 \times 1{\cdot}12 \;=\; 44{\cdot}8 \text{ mols}$$
$$V_n = L_n + D = 44{\cdot}8 + 40 \;=\; 84{\cdot}8 \text{ mols}$$
$$V_m = L_m - W = 144{\cdot}8 - 60 = \;84{\cdot}8 \text{ mols}$$
$$L_m = L_n + F = 44{\cdot}8 + 100 = 144{\cdot}8 \text{ mols}$$
$$\frac{L_m}{W} = \frac{144{\cdot}8}{60} = 2{\cdot}41$$

7. *Check on Minimum Reflux Ratio.*

$$x_n = \frac{x_d}{(\alpha - 1)R_m} \text{ for components lighter than heavy key}$$

For $n$-$C_4$ $$x_n = \frac{0{\cdot}975}{(2{\cdot}38 - 1)1{\cdot}12} = 0{\cdot}630$$

and $n$-$C_5$ $$x_n = 1 - 0{\cdot}630 = 0{\cdot}370$$

*Temperature Check for Upper Pinch*, $\Sigma K x_n = 1{\cdot}0$

$$\Sigma K \,.\, x_n = 1{\cdot}62 \times 0{\cdot}630 + 0{\cdot}68 \times 0{\cdot}370 = 1{\cdot}273$$

Upper pinch temperature of 202°F is incorrect.

Try $T_n = 180$°F.

| *Component* | $K_{180}$ | $\alpha$ | $x_n$ | $Kx_n$ |
|---|---|---|---|---|
| $C_4$ | 1·35 | 2·55 | 0·562 | 0·759 |
| $C_5$ | 0·53 | 1 | 0·438 | 0·233 |
| | | | 1 | 0·992 |

This is sufficiently near, so take the upper pinch temperature as 180°F

$$\therefore \qquad r_n = \frac{0{\cdot}562}{0{\cdot}438} = 1{\cdot}282$$

Since there is no component lighter than light key $\Sigma b_n x_n = 0$.

In the lower pinch $x_m = \dfrac{\alpha_{AB} \,.\, x_w}{(\alpha_{AB} - \alpha)L_m/W}$ from equation 18.139.

| *Component* | $K_{246}$ | $\alpha$ | $\alpha_{AB} - \alpha$ | $x_m$ | $Kx_m$ |
|---|---|---|---|---|---|
| $C_4$* | 2·2 | 2 | | 0·384 | 0·845 |
| $C_5$ | 1·1 | 1 | 1·0 | 0·305 | 0·335 |
| $C_6$ | 0·51 | 0·464 | 1·536 | 0·153 | 0·078 |
| $C_7$ | 0·28 | 0·254 | 1·746 | 0·158 | 0·044 |
| | | | | 1 | 1·302 |

* $x_m$ by difference.

There is no temperature check so that $T_m \neq 246$.

Try $T_m = 210$°F.

| *Component* | $K_{210}$ | $\alpha$ | $\alpha_{AB} - \alpha$ | $x_m$ | $K\,.\,x_m$ | $b_m$ | $b_m\alpha_m x_m$ |
|---|---|---|---|---|---|---|---|
| $C_4$* | 1·70 | 2·30 | — | 0·428 | 0·729 | | |
| $C_5$ | 0·74 | 1· | 1·30 | 0·270 | 0·200 | | |
| $C_6$ | 0·35 | 0·47 | 1·83 | 0·148 | 0·052 | 1·22 | 0·085 |
| $C_7$ | 0·17 | 0·23 | 2·07 | 0·154 | 0·026 | 0·91 | 0·032 |
| | | | | 1 | 1·007 | | 0·117 |

* $x_m$ by difference.

This is sufficiently near so $T_m = 210$°F.

Then $r_m = \dfrac{0\cdot428}{0\cdot270} = 1\cdot586 \qquad \alpha\dfrac{r_m}{r_n} = \dfrac{1\cdot586}{1\cdot282} = 1\cdot235$ and $\psi = \dfrac{1}{1 - 0\cdot117} = \dfrac{1}{0\cdot883} = 1\cdot132$

Hence $R_m$ is not quite equal to 1·12.

8. Now try $R_m = 1\cdot08$

$$L_n = 40 \times 1\cdot08 = 43\cdot2$$
$$V_n = 40 + 43\cdot2 = 83\cdot2 = V_m$$
$$L_m = 143\cdot2$$

Take $T_n = 180$°F as before

| *Component* | $K_{180}$ | $\alpha$ | $x_n$ | $Kx_n$ | |
|---|---|---|---|---|---|
| $C_4$ | 1·35 | 2·55 | 0·582 | 0·785 | |
| $C_5$ | 0·53 | 1 | 0·418 | 0·221 | |
| | | | 1 | 1·006 | Checks |

$$\therefore \qquad r_n = \frac{0\cdot582}{0\cdot418} = 1\cdot393$$

Take $T_m = 210$°F as before

| *Component* | $K_{210}$ | $\alpha$ | $\alpha_{AB} - \alpha$ | $x_m$ | $Kx_m$ | $b_m$ | $b_m\,.\,\alpha_m\,.\,x_m$ |
|---|---|---|---|---|---|---|---|
| $C_4$* | 1·70 | 2·30 | — | 0·424 | 0·721 | | |
| $C_5$ | 0·74 | 1 | 1·30 | 0·272 | 0·201 | | |
| $C_6$ | 0·35 | 0·47 | 1·83 | 0·149 | 0·052 | 1·22 | 0·085 |
| $C_7$ | 0·17 | 0·23 | 2·07 | 0·155 | 0·026 | 0·91 | 0·034 |
| | | | | 1 | 1 | | 0·119 |

* $x_m$ by difference.

∴ $$r_m = \frac{0.424}{0.272} = 1.558 \qquad \alpha \frac{r_m}{r_n} = \frac{1.558}{1.393} = 1.12$$

and $$\psi = \frac{1}{1 - 0.17} = 1.13$$

∴ $R_m = 1.08$ is near enough.

Since there are no components lighter than the light key, we should expect the lower pinch to be near the feed plate as it is, but the general method of taking the pinch temperature as one-third and two-thirds up the column was used above.

The small change in $R$ from 1·12 – 1·08 gives a change in $r_n$ but very little change in $r_m$. We see that the first estimation for $R_m$ of 1·12 based on the equation 18.137 for locating the upper pinch composition is nearly correct but that it gives the wrong pinch composition.

***Minimum Reflux Ratio $R_m$, using Underwood's Method.*** For conditions where the relative volatilities remain constant, UNDERWOOD(33) has developed the following two equations from which $R_m$ may be calculated.

$$\frac{\alpha_A x_{fA}}{\alpha_A - \theta} + \frac{\alpha_B x_{fB}}{\alpha_B - \theta} + \frac{\alpha_C x_{fC}}{\alpha_C - \theta} + \ldots = 1 - q \qquad \ldots.(18.141)$$

and $$\frac{\alpha_A x_{dA}}{\alpha_A - \theta} + \frac{\alpha_B x_{dB}}{\alpha_B - \theta} + \frac{\alpha_C x_{dC}}{\alpha_C - \theta} + \ldots = R_m + 1 \qquad \ldots.(18.142)$$

where, $x_{fA}$, $x_{fB}$, $x_{fC}$, $x_{dA}$, $x_{dB}$, $x_{dC}$, etc., are the mol fractions of components $A$, $B$, $C$, etc., in the feed and distillate, $A$ being the light and $B$ the heavy key,

$q$ is the ratio of the heat required to vaporise 1 mol of the feed to the molar latent heat of the feed, as on p. 620,

$\alpha_A$, $\alpha_B$, $\alpha_C$, etc. are the volatilities with respect to the least volatile component, and

$\theta$ is the root of equation 18.141, which lies between the values of $\alpha_A$ and $\alpha_B$.

**Example.** Suppose a mixture of hexane, heptane, and octane to be separated to give products as shown in the table. What will be the value of the minimum reflux ratio, if the feed is liquid at its boiling point?

| *Component* | *Feed* |  | *Product* |  | *Bottoms* |  | *Relative volatility* |
|---|---|---|---|---|---|---|---|
|  | *F* mols | $x_f$ | *D* mols | $x_d$ | *W* mols | $x_w$ |  |
| C. Hexane | 40 | 0·40 | 40 | 0·534 | 0 | 0 | 2·70 |
| B. Heptane | 35 | 0·35 | 34 | 0·453 | 1 | 0·04 | 2·22 |
| A. Octane | 25 | 0·25 | 1 | 0·013 | 24 | 0·96 | 1·0 |

**Solution.**

From equation 18.141 with $q = 1$,

$$\frac{2.70 \times 0.40}{2.70 - \theta} + \frac{2.22 \times 0.35}{2.22 - \theta} + \frac{1 \times 0.25}{1 - \theta} = 0$$

The required value of $\theta$ must satisfy the relation $\alpha_A < \theta < \alpha_B$, i.e. $1.0 < \theta < 2.22$.

Try 1·15: then $\sum \frac{\alpha \,.\, x_f}{\alpha - \theta} = -0.243$

Try 1·17: then $\sum \frac{\alpha \,.\, x_f}{\alpha - \theta} = -0.024$

This is near enough, so that from equation 18.142,

$$\frac{2{\cdot}70 \times 0{\cdot}534}{2{\cdot}70 - 1{\cdot}17} + \frac{2{\cdot}22 \times 0{\cdot}453}{2{\cdot}22 - 1{\cdot}17} + \frac{1{\cdot}00 \times 0{\cdot}013}{1{\cdot}00 - 1{\cdot}17} = 1{\cdot}827$$

$$\therefore \qquad \underline{R_m = 0{\cdot}827}$$

### Number of Plates at Total Reflux

The number of plates required for the desired separation under conditions of total reflux can be found by applying FENSKE's equation (18.39) to the two key components.

Thus

$$n + 1 = \frac{\log \left(\frac{x_A}{x_B}\right)_d \cdot \left(\frac{x_B}{x_A}\right)_w}{\log (\alpha_{AB})_{\text{av.}}} \qquad \ldots .(18.143)$$

**Example.** For the separation of hexane, heptane, and octane as in the previous example, determine the number of theoretical plates required.

**Solution.**

$$n+1 = \frac{-\log \left[\frac{0{\cdot}453}{0{\cdot}013}\right] \times \left[\frac{0{\cdot}960}{0{\cdot}04}\right]}{-\log 2{\cdot}22} = \frac{\log 835}{\log 2{\cdot}22}$$

$$= 8{\cdot}5.$$

Thus, the minimum number of plates = $\underline{\underline{7{\cdot}5, \text{ say } 8.}}$

### Relation between Reflux Ratio and Number of Plates

GILLILAND[24] has given an empirical relation between the reflux ratio $R$ and the number of plates $n$, in which only the minimum reflux ratio $R_m$ and the number of plates at total reflux $n_m$ are required. This is shown in Fig. 18.58, where $\frac{R - R_m}{R + 1}$ is plotted against the group $\frac{(n + 1) - (n + 1)_m}{n + 2}$

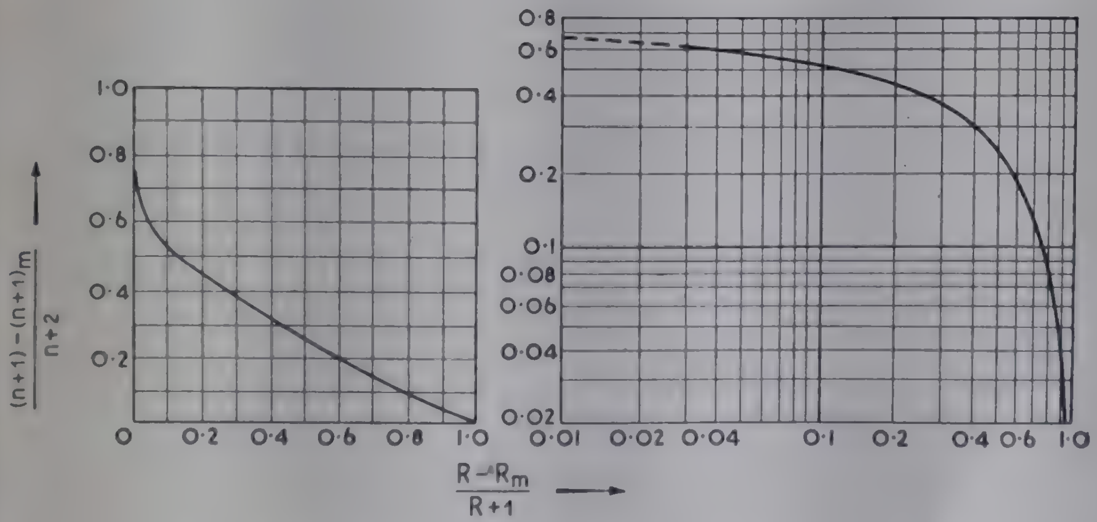


Fig. 18.58. Relation between reflux ratio and number of plates

**Example.** Using the example above we can set out the change in $n$ with $R$ as follows:

Note: $R_m = 0{\cdot}83$ and $(n+1)_m = 8{\cdot}5$.

| *Value* of $R$ | $\dfrac{R - R_m}{R + 1}$ | $\dfrac{(n+1) - (n+1)_m}{n+2}$ | $n$ |
|---|---|---|---|
| 1 | $\dfrac{0{\cdot}17}{2} = 0{\cdot}085$ | 0·55 | 19 |
| 2 | $\dfrac{1{\cdot}17}{3} = 0{\cdot}390$ | 0·32 | 11·8 |
| 5 | $\dfrac{4{\cdot}17}{6} = 0{\cdot}695$ | 0·15 | 9·1 |
| 10 | $\dfrac{9{\cdot}17}{11} = 0{\cdot}833$ | 0·08 | 8·2 |

This illustration shows that the number of plates falls off rapidly at first, but slowly later, and a value of $R$ of nearly two is probably the most economic.

## AZEOTROPIC AND EXTRACTIVE DISTILLATION

In the processes so far considered, the vapour becomes steadily richer in the more volatile component on successive plates. There are two types of mixtures where this steady increase in the concentration of the more volatile component either does not take place, or else takes place so slowly that an uneconomical number of plates are required. If, for instance, we concentrate a mixture of ethyl alcohol and water, the concentration of the alcohol steadily increases until we reach 96% by weight, when the composition of the vapour equals that of the liquid, and no further enrichment occurs. This mixture is called an azeotrope, and cannot be separated by straightforward distillation. Such a condition is shown in the $y$–$x$ curves of Fig. 18.2, where it will be seen that the equilibrium curve crosses the diagonal, indicating an azeotrope. A large number of azeotropic mixtures have been found, some of which are of great industrial importance, e.g. water–nitric acid; water-hydrochloric acid; water–many alcohols. An example of the second type of problem is the separation of *n*-heptane from methyl cyclohexane. Here the relative volatility is only 1·08 and a large number of plates are required to achieve separation.

The principle of azeotropic and of extractive distillation lies in the addition of a new substance to the mixture so as to increase the relative volatility of the two key components, and thus make separation relatively easy. BENEDICT and RUBIN(31) have defined these two processes in the following way. In azeotropic distillation the substance added forms an azeotrope with one or more of the components in the mixture, and as a result is present on most of the plates of the column in appreciable concentration. With extractive distillation the substance added is relatively non-volatile compared with the components to be separated, and it is therefore fed continuously near the top of the column. This extractive agent runs down the column as reflux and is present in appreciable concentration on all the plates.

**Azeotropic Distillation. Ethyl Alcohol–Water**

Young(2), about 1902, found that if benzene was added to the alcohol–water azeotrope; then a ternary azeotrope was formed with a boiling point of 64·85°C, i.e., less than that of the binary azeotrope (78·15°C). The industrial production of ethyl alcohol using this principle has been described by Guinot and Clark(22), and the general arrangement of the plant is as indicated in Fig. 18.59. This requires the use of three fractionating columns *A*, *C*, and *D*, and a continuous two-phase liquid separator *B*.

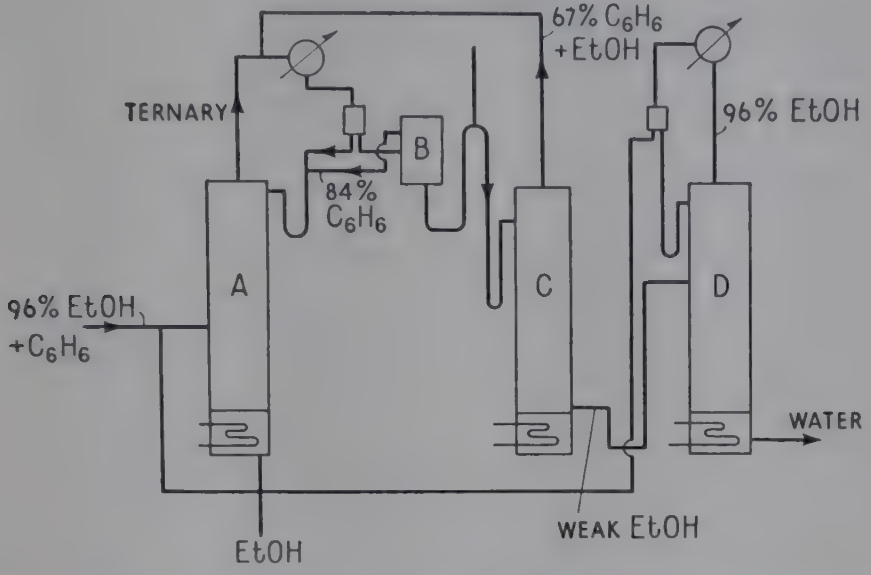


Fig. 18.59. Arrangement of plant for azeotropic distillation of ethanol-water mixtures, using benzene as the extractive agent

The 96% alcohol–water azeotrope together with the required amount of benzene (or other entrainer) is fed to column *A*. From the bottom of this column is obtained pure alcohol, whilst from the top a ternary azeotrope is obtained which, on condensation, is passed to the separator *B*. In this separator two liquid phases are formed, the upper one being richer in benzene and the lower in alcohol, with the compositions shown below. The upper layer is

*Concentrations in alcohol–water azeotropic distillation*

| | *Column A Vapour %* | *Decanter B* | | *Column C Vapour %* |
|---|---|---|---|---|
| | | *Upper layer* 84% *of volume* | *Lower layer* 16% *of volume* | |
| Alcohol | 18·5 | 14·5 | 53 | 32·4 |
| Benzene | 74·1 | 84·5 | 11 | 67·6 |
| Water | 7·4 | 1 | 36 | |
| B. Pt. °C | 64·85 | | | 68·2 |

returned to column *A* as part of the reflux and the lower layer is passed to column *C*, where the benzene is obtained as a benzene–alcohol azeotrope. The weak alcohol–water mixture obtained from the bottom of column *C* is then passed to column *D* where water is removed at the base and the alcohol is recovered at the top as the 96% azeotrope, which is then passed as additional feed to column *A*. With this system column *A* is the main azeotropic column whilst column *C* is used to recover the entrainer and column *D* to recover any remaining alcohol.

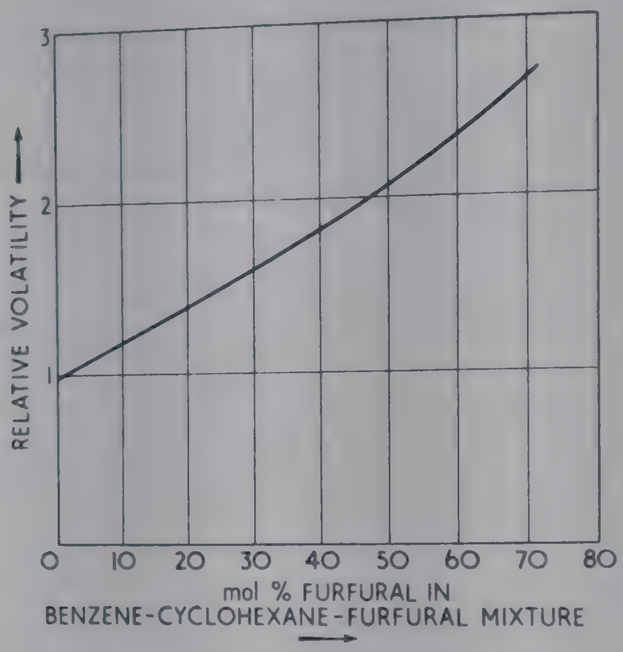


Fig. 18.60. Effect of furfural as extractive agent on relative volatilities of 50% benzene-cyclohexane mixture

The successful operation of this system relies on three main factors. First, the water is removed as a ternary azeotrope from the first column, allowing the alcohol to be stripped and removed at the bottom. Secondly, the ternary azeotrope separates easily into two layers in the decanter, thus enabling the bulk of the entrainer to be returned to the first column. Thirdly, the heat requirement to vaporise the entrainer is not an appreciable fraction of the total heat used for the separation. NORMAN(32) has given equilibrium data for this system and shown how the number of plates required may be determined. The relative volatilities of pairs of components are plotted against the ratio of their concentrations in the liquid phase. Then the composition of the vapour in equilibrium with a given liquid composition can be found.

## Extractive Distillation

For a non-ideal binary mixture we may express the partial pressure in the form

$$P_A = \gamma_A P_A^\circ \,.\, x_A \qquad \ldots.(18.144a)$$

$$P_B = \gamma_B P_B^\circ \,.\, x_B \qquad \ldots.(18.144b)$$

where $\gamma_A$ and $\gamma_B$ are the activity coefficients for the two components. The relative volatility $\alpha$ may thus be written,

$$\alpha = \frac{P_A}{P_B} \cdot \frac{x_B}{x_A} = \frac{\gamma_A}{\gamma_B} \frac{P_A^\circ}{P_B^\circ} \qquad \ldots .(18.145)$$

The solvent added to the mixture in extractive distillation differentially affects the activities of the two components, and hence $\alpha$. This is shown below for the addition of furfural to a mixture of benzene and cyclohexane. (See Fig. 18.60).

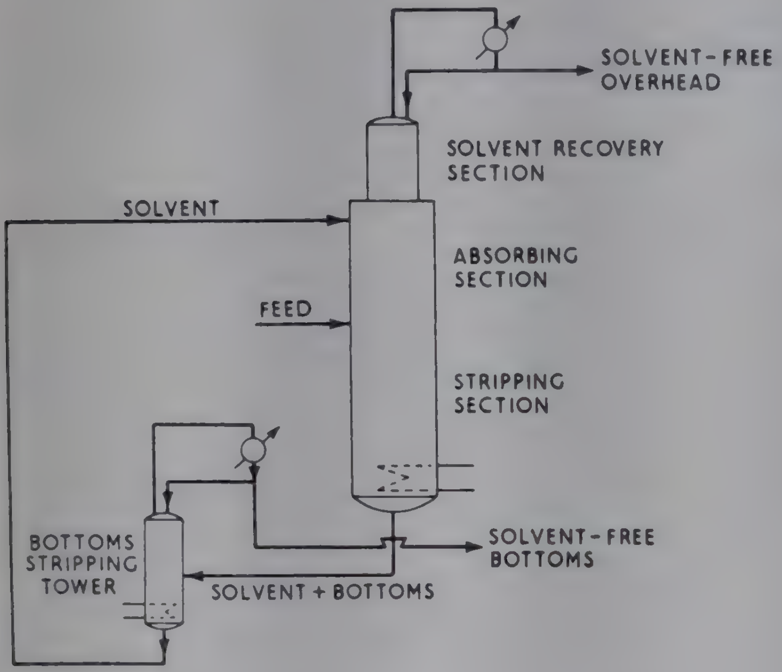


Fig. 18.61. An extractive distillation unit

| Mol % furfural | 0 | 20 | 40 | 50 | 60 | 70 |
|---|---|---|---|---|---|---|
| Relative Volatility $\alpha$ | 0·98 | 1·38 | 1·86 | 2·07 | 2·35 | 2·70 |

A general arrangement for an extractive distillation unit is shown in Fig. 18.61. The extracting solvent is fed in near the top of the main column and, being relatively non-volatile, flows down as reflux. The few plates above the main column prevent any solvent rising into the top product. The bottoms from the main column are fed to a second column where they are freed from solvent, which is then recirculated to the main column.

The solvent to be used will be selected on the basis of, (*a*) selectivity, (*b*) volatility, (*c*) ease of separation from the top and bottom products, and (*d*) the cost. The selectivity is most easily assessed by determining the effect on the relative volatility of the two key components of addition of the solvent. The more volatile the solvent, the greater the percentage of solvent in the vapour, and the poorer the separation for a given heat consumption in the boiler. It is important to note that the solvent must not form an azeotrope with any of the components. Some of the problems of selecting the solvent are discussed by SCHEIBEL(38), who points out that use may be made of the fact that, when two compounds show deviations from Raoult's law, then one of these compounds shows the same type of deviation with any member of the homologous series of the other component. Thus the azeotrope acetone (B.Pt. 56·4)- methanol (B.Pt. 64·7) has 20 mol % acetone and boils at 55·7°C, i.e. less than the boiling point of either component. Then any member of the series ethanol (78·3), propanol (97·2), water (100), butanol (117.8) may be used as an extractive agent, or in the series of ketones, methyl *n*-propyl ketone (102), methyl isobutyl ketone (116). The advantages of using a solvent from the alcohol series is that the more volatile acetone will be taken overhead, and water would have the advantage of cheapness. PRATT(35) has given details of a method of calculation for extractive distillation using the system acetonitrile-trichlorethylene-water, as an example.

## STEAM DISTILLATION

If the material to be distilled has a high boiling point and is almost or completely immiscible with water, then a process of steam distillation may be used. Steam is blown directly into the boiler of a batch still, containing a liquid such as aniline or nitrobenzene which is to be purified. Some of the steam will condense in heating the liquid to its boiling point and, since aniline (say) is virtually immiscible with water, two liquid phases will form in the still. The vapour will consist of steam and aniline, the proportion of aniline being given by the relation:

$$\frac{m_A}{m_B} = \frac{P_A}{P_B} \cdot \frac{M_A}{M_B} \qquad \ldots .(18.147)$$

where $m_A$, $m_B$ are the masses of aniline ($A$) and steam ($B$), respectively,
$M_A$, $M_B$ are the molecular weights of aniline and steam, and
$P_A$, $P_B$ are the vapour pressures of aniline and steam at the boiling point of the mixture.

By applying the phase rule, we can see that there will be only one degree of freedom, so that either the temperature or the pressure may be fixed, the water and other liquid each exerting a partial pressure equal to its vapour pressure at the boiling point of the mixture. This relation will only be approximately true if the material is slightly miscible with water.

A convenient method for calculating the temperature and composition of the vapour is by the use of the diagram shown in Fig. 18.62, due to HAUSBRAND. Here the vapour pressure of water is plotted as $P - P_B$, where $P$ is the total pressure, and the vapour pressures of a number of other materials are plotted directly against the temperature. The intersection of the water curve with that of the material being distilled will give the temperature of distillation, and the molar ratio of water to organic liquid will be given by $\dfrac{P - P_A}{P_A}$. In the diagram, curves for water are given for total pressures of 760, 300 and 70 mm of mercury. Thus, if nitrobenzene is distilled at atmospheric pressure with live saturated steam, the boiling point will be about 99°C and the mass ratio of water to nitrobenzene in the vapour will be $\frac{760 - 20}{20} \cdot \frac{18}{123} = 5{\cdot}4$.

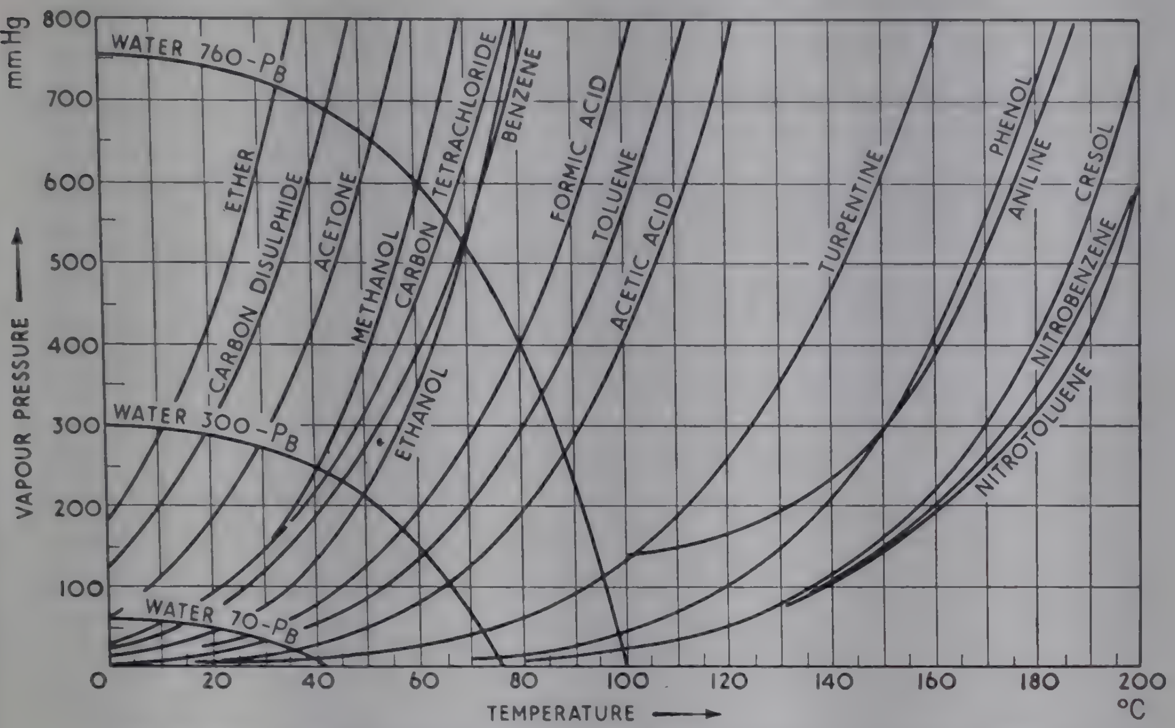


Fig. 18.62. Vapour pressure curves for calculations on steam distillation

The attraction of the method is that it enables a high boiling material to be distilled with low-pressure steam at a relatively low temperature so that decomposition is unlikely to occur. If superheated steam is passed into liquid which is maintained at its boiling point by an external source of heat, distillation can be effected at a low temperature, but it is not possible to give a simple relation for the composition of the vapour. It is possible in this manner to prevent condensation of steam in the still. In any operation of this kind, it is important that the separation of the material being distilled from the water should be easy.

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## LIST OF SYMBOLS USED IN CHAPTER 18

| | | |
|---|---|---|
| $A$ | Interfacial area for transfer | $L^2$ |
| $A'$ | Interfacial area between bubbles and liquid | $L^2$ |
| $a$ | Interfacial surface per unit volume of column | $L^{-1}$ |
| $B$ | Coefficient in equation 18.62 | $LT^{-1}$ |
| $B'$ | Constant in equation 18.96 | — |
| $B''$ | Constant in equation 18.97 | — |
| $b_m, b_n$ | Factors in equation 18.140 | — |
| $C$ | Concentration in mols/unit volume | $ML^{-3}$ |
| $C'$ | Coefficient in equation 18.60 | $M^{-1/2}L^{5/2}T^{-1}$ |
| $C_D$ | Coefficient of discharge for slot | — |
| $c$ | Constant in vapour-liquid equilibrium relations | — |
| $D$ | Mols or mass of product per unit time | $MT^{-1}$ |
| $D_b$ | Mols of product in batch distillation | $M$ |
| $D_L$ | Diffusivity in the liquid phase | $L^2T^{-1}$ |
| $D_v, D$ | Diffusivity in the vapour phase | $L^2T^{-1}$ |

| | | |
|---|---|---|
| $d$ | Bubble diameter | L |
| $d_c$ | Column diameter | L |
| $d_r$ | Diameter of ring | L |
| $E$ | Average Murphree plate efficiency | — |
| $E_m$ | Local Murphree plate efficiency | — |
| $E'$ | Rate of change in composition with height for unit driving force | $L^{-1}$ |
| $e$ | Fractional voidage of packing | — |
| $F$ | Mols or mass of feed per unit time | $MT^{-1}$ |
| $F_w$ | BOLLES' factor for flow over weir (equation 18.71) | $L^{-1/3}T^{2/3}$ |
| $F''$ | Mols of feed | M |
| $G$ | Mass or mols of vapour per unit time | $MT^{-1}$ |
| $G'$ | Mols of vapour per unit time and unit cross-section | $ML^{-2}T^{-1}$ |
| $g$ | Acceleration due to gravity | $LT^{-2}$ |
| $H$ | Enthalpy per mol or unit mass | $\theta$ |
| $H^L$ | Enthalpy per mol or unit mass of liquid | $\theta$ |
| $H^V$ | Enthalpy per mol or unit mass of vapour | $\theta$ |
| $H_d'$ | $H_d^L + \dfrac{Q_C}{D}$ | $\theta$ |
| $H'_w$ | $H_w^L - \dfrac{Q_B}{W}$ | $\theta$ |
| $\mathbf{H}$ | Height of transfer unit | L |
| $\mathbf{H}_g$ | Height of transfer unit—gas film | L |
| $\mathbf{H}_{og}$ | Height if transfer unit—overall (gas concentrations) | L |
| $\mathbf{H}_l$ | Height of transfer unit—liquid film | L |
| $\mathbf{H}_{ol}$ | Height of transfer unit—overall (liquid concentration) | L |
| $\mathscr{H}'$ | Henry's constant ($P_A/x_A$) | $ML^{-1}T^{-2}$ |
| $h$ | Height of slot | L |
| $h_d$ | Loss in head at bottom of downcomer | L |
| $h_g$ | Drop in head across plate | L |
| $h_L$ | Effective submergence | L |
| $h_v$ | Loss in head of vapour passing through plate | L |
| $h_r$ | Loss in head due to contraction at cap riser | L |
| $h_{rc}$ | Loss in head due to riser and reversal inside cap | L |
| $h_s$ | Loss in head through slot (i.e. slot opening) | L |
| $h_{ss}$ | Static submergence (top of slot to weir) | L |
| $h_w$ | Height of exit weir | L |
| $h_{ow}$ | Height of crest over weir | L |
| $\Delta h_e$ | Drop in head at exit weir | L |
| $\Delta h_i$ | Drop in head at inlet weir | L |
| $\Delta h_o$ | Drop in head at zero seal | L |
| $j_d$ | $j$-factor for mass transfer | — |
| $K$ | Equilibrium constant ($=y/x$) | — |
| $K_g$ | Overall mass transfer coefficient | $LT^{-1}$ |
| $K_g'$ | Overall mass transfer coefficient (mols/unit time-unit area-unit mol fraction driving force) | $ML^{-2}T^{-1}$ |
| $K_l$ | Overall mass transfer coefficient | $LT^{-1}$ |
| $K_l'$ | Overall mass transfer coefficient | $ML^{-2}T^{-1}$ |
| $k_g, k_g'$ | Film coefficients corresponding to $K_g$, $K_g'$, above | $LT^{-1}$, $ML^{-2}T^{-1}$ |
| $k_l, k_l'$ | Film coefficients corresponding to $K_L$, $K_L'$ | $LT^{-1}$, $ML^{-2}T^{-1}$ |
| $L$ | Liquid flow in mass or mols/unit time | $MT^{-1}$ |
| $L'$ | Liquid flow, mols/unit time-unit area | $ML^{-2}T^{-1}$ |
| $L_b$ | Mols of liquid in batch distillation | M |
| $L_p$ | Liquid rate per unit periphery | $L^2T^{-1}$ |

| | | |
|---|---|---|
| $M$ | Molecular weight | — |
| | *or* difference stream below feed plate | $\mathrm{MT^{-1}}$ |
| $M_m$ | Mean molecular weight | — |
| $m$ | Mass of material | M |
| | *or* gradient of equilibrium curve | — |
| $N$ | Molar rate of transfer per unit area of interface | $\mathrm{ML^{-2}T^{-1}}$ |
| | *or* difference stream above feed plate | $\mathrm{MT^{-1}}$ |
| $\mathbf{N}$ | Number of transfer units | — |
| $\mathbf{N}_g$, $\mathbf{N}_{og}$ | Number of gas film and overall gas transfer units | — |
| $\mathbf{N}_l$, $\mathbf{N}_{ol}$ | Number of liquid film and overall liquid transfer units | — |
| $n$ | Number of (theoretical) plates | — |
| $n_m$ | Number of plates at total reflux | — |
| $n_p$ | Number of actual plates | — |
| $P$ | Total pressure | $\mathrm{ML^{-1}T^{-2}}$ |
| $P_A$, $P_B$ | Partial pressure of $A$, $B$ | $\mathrm{ML^{-1}T^{-2}}$ |
| $P_A^{\circ}$ | Vapour pressure of $A$ | $\mathrm{ML^{-1}T^{-2}}$ |
| $p$ | Constant in equation 18.99 | — |
| $Q$ | Quantity of heat | $\mathrm{M\theta}$ |
| $Q_B$ | Heat supplied to boiler for bottom product $W$ | $\mathrm{M\theta T^{-1}}$ |
| $Q_C$ | Heat removed in condenser for product $D$ | $\mathrm{M\theta T^{-1}}$ |
| $Q_R$ | Heat to still in batch distillation to provide reflux | $\mathrm{M\theta}$ |
| $Q'$ | Liquid rate | $\mathrm{L^3T^{-1}}$ |
| $q$ | Heat to vaporise one mol of feed divided by molar latent heat | — |
| $q_C$, $Q_C/D$ | | $\theta$ |
| $R$ | Reflux ratio | — |
| $R_m$ | Minimum reflux ratio | — |
| $R'$ | Resistance force per unit area, as used in Chapter 11 | $\mathrm{ML^{-1}T^{-2}}$ |
| $\mathbf{R}$ | Universal gas constant | $\mathrm{L^2T^{-2}\theta^{-1}}$ |
| $r$ | Radius of an orifice | L |
| $r_n$, $r_m$, $r_f$ | Ratio of concentrations of key components on plates $m$, $n$ and in the feed | — |
| $S$ | Mols of material in the still | M |
| $S_m$ | Liquid seal $= (h_{ss} + h_{ow})$ | L |
| $s$ | Cross-sectional area of column | $\mathrm{L^2}$ |
| $T$ | Absolute temperature | $\theta$ |
| $t$ | Time | T |
| $u$ | Vapour velocity | $\mathrm{LT^{-1}}$ |
| $u_a$ | Velocity of rise of bubble | $\mathrm{LT^{-1}}$ |
| $u_d$ | Velocity of liquid in downcomer | $\mathrm{LT^{-1}}$ |
| $u_r$ | Velocity through riser | $\mathrm{LT^{-1}}$ |
| $u_s$ | Vapour velocity through slot | $\mathrm{LT^{-1}}$ |
| $V$ | Vapour flow in mols or mass per unit time | $\mathrm{MT^{-1}}$ |
| $V'$ | Vapour as bubbles | M |
| $V''$ | Mols of vapour formed | M |
| $v_s$ | Volumetric rate of flow of vapour through slot | $\mathrm{L^3T^{-1}}$ |
| $W$ | Bottom product in mols or mass per unit time | $\mathrm{MT^{-1}}$ |
| $w$ | Slot width or width of weir | L |
| $x$ | Mol fraction of a component in the liquid phase | — |
| $y$ | Mol fraction of a component in the gas phase | — |
| $Z$ | Height of packed column | L |
| $Z_p$ | Plate spacing | L |
| $Z_p'$ | Depth of liquid in the downcomer | L |
| $Z_t$ | Height of equivalent theoretical plate | L |
| $z$ | Distance | L |
| $\alpha$ | Relative volatility or volatility relative to heavy key | — |
| $\alpha_{AB}$ | Volatility of $A$ relative to $B$ | — |

| | | |
|---|---|---|
| $\gamma$ | Activity coefficient | — |
| $\lambda$ | Latent heat per mol or per unit mass | $\theta$ |
| $\mu_L$ | Viscosity of liquid | $ML^{-1}T^{-1}$ |
| $\mu_v$, $\mu$ | Viscosity of vapour | $ML^{-1}T^{-1}$ |
| $\rho_L$ | Density of liquid | $ML^{-3}$ |
| $\rho_v$, $\rho$ | Density of vapour | $ML^{-3}$ |
| $\sigma$ | Surface tension | $MT^{-2}$ |
| $\theta$ | Root of equation 18.142 | — |
| $\phi$ | Intercept of operating line on $Y$-axis $\dfrac{x_d}{R+1}$ | — |
| $\psi$ | $r_m/r_n$ at minimum reflux | — |
| $\omega$ | Fraction of packing wetted | — |
| *Re.* | Reynolds Group | — |
| *Sc.* | Schmidt Group | — |

Suffixes:

1, 2 Inlet, outlet
*A, B, C, D* Materials *A, B, C, D*
*b* Bottom
*c* Intersection of equilibrium and operating lines at minimum reflux
*d* Top product
*e* Equilibrium
*f* Feed
*fs* feed at its boiling point
*g* azeotrope
*h* component heavier than heavy key
*i* interface
*m, n* Plates *m, n* below and above feed plate
*q* intersection of operating lines
*s* still
*t* top
*w* Bottom product

## CHAPTER 19

# *Absorption of Gases*

THE removal of one or more selected components from a mixture of gases by absorption into a suitable liquid is the second major operation of chemical engineering that is based on interphase mass transfer controlled largely by rates of diffusion. Thus, acetone can be recovered from an acetone-air mixture by passing the gas stream into water in which the acetone dissolves while the air passes out. Similarly, ammonia may be removed from an ammonia-air mixture by absorption in water. In each of these examples the process of absorption of the gas in the liquid can be treated as a physical process, the chemical reaction having no appreciable effect. However, oxides of nitrogen are absorbed in water to give nitric acid, or when carbon dioxide is absorbed in a solution of sodium carbonate, a chemical reaction occurs, the nature of which influences the actual rate of absorption. Absorption processes are therefore conveniently divided into two groups, those in which the process is solely physical and those where a chemical reaction is occurring. In considering the design of equipment to achieve gas absorption, the main requirement is that the gas be brought into intimate contact with the liquid, and the effectiveness of the equipment will largely be determined by the success with which it promotes contact between the two phases. The general form of equipment is similar to that described earlier for distillation (Chapter 18), and packed and plate towers are generally used for large installations. The method of operation, as will be seen later, is not the same. In absorption, the feed is a gas and is introduced at the bottom of the column, and the solvent is fed to the top, as a liquid; the absorbed gas and solvent leave at the bottom, and the unabsorbed components leave as gas from the top. The essential difference between distillation and absorption is that in the former the vapour has to be produced in each stage by partial vaporisation of the liquid which is therefore at its boiling point, whereas in absorption the liquid is well below its boiling point. In distillation there is a diffusion of molecules in both directions so that for an ideal system we have equimolecular counter-diffusion, but in absorption gas molecules are diffusing into the liquid, and the movement in the reverse direction is negligible (Vol. 1, Chapter 7).

## CONDITIONS OF EQUILIBRIUM BETWEEN LIQUID AND GAS

The two phases when brought into contact tend to reach equilibrium. Thus, water in contact with air evaporates until the air is saturated with water vapour, and the air is absorbed by the water until it becomes saturated with the individual gases. In any mixture of gases, the degree to which each gas is absorbed is determined by its partial pressure. At a given temperature and concentration, each dissolved gas exerts a definite partial pressure. We may consider three types of gases from this aspect: a very soluble one (ammonia), a moderately soluble one (sulphur dioxide), and a slightly soluble one (oxygen). The figures in Table 19.1 show the concentrations in grams per 1,000 grams of water that are required to develop a partial pressure of 10, 50, 100, 200 and 500 mm of mercury at 30°C.

TABLE 19.1. *Partial pressures of solutions of gases*

| Partial pressure in mm of Hg | 10 | 50 | 100 | 200 | 500 |
|---|---|---|---|---|---|
| Ammonia | 11 | 50 | 93 | 160 | 315 |
| Sulphur dioxide | 1·9 | 6·8 | 12 | 24·4 | 56 |
| Oxygen | | | 0·08 | 0·13 | 0·33 |

It is seen that a slightly soluble gas requires a much higher partial pressure of the gas in contact with the liquid to give a solution of a given concentration. Conversely, with a very soluble gas a given concentration in the liquid phase is obtained with a lower partial pressure in the vapour phase. At 20°C a solution of 4 grams of sulphur dioxide per 1,000 grams of water exerts a partial pressure of 20 mm of Hg. If therefore we have a gas in contact with this solution with a partial pressure of $SO_2$ greater than 20 mm of Hg, sulphur dioxide will be absorbed. The most concentrated solution that can be obtained is one in which the partial pressure of the solute gas is equal to the partial pressure of the gas in the gas phase. These equilibrium conditions fix the limits of operation of an absorption unit. Thus, in an ammonia-air mixture containing 13·1% of ammonia, the partial pressure of the ammonia is 100 mm and the maximum concentration of the ammonia in the water at 30°C is 93 grams per 1,000 grams of water.

Whilst the solubility of a gas is not substantially affected by the total pressure in the system for pressures up to about 5 atmospheres, it is important to note that the solubility falls with a rise of temperature. Thus, for a concentration of 25% by weight of ammonia in water, the equilibrium partial pressure of the ammonia is 227 mm at 20°C and 352 mm at 30°C. In many instances the absorption is accompanied by the evolution of heat, and it is therefore necessary to fit coolers to the equipment to keep the temperature sufficiently low for an adequate degree of absorption to be obtained.

For dilute concentrations of most gases, and over a wide range for some gases, the equilibrium relationship is given by Henry's law. This law, as used in Chapter 18, can be written as,

$$P_A = \mathscr{H} C_A \qquad \ldots.(19.1)$$

where, $P_A$ is the partial pressure of the component $A$ in the gas phase,

$C_A$ is the concentration of the component in the liquid, and

$\mathscr{H}$ is Henry's constant.

## THE MECHANISM OF ABSORPTION
## THE TWO-FILM THEORY

The most useful concept of the process of absorption is given by the two-film theory due to WHITMAN$^{(4)}$. According to this theory, material is transferred in the bulk of the phases by convection currents, and concentration differences are regarded as negligible except in the vicinity of the interface between the phases. On either side of this interface it is supposed that the currents die out and that there exists a thin film of fluid through which the transfer is effected solely by molecular diffusion. This film will be slightly thicker than the laminar sub-layer, because it offers a resistance equivalent to that of the whole boundary layer. According to Fick's law (equation 7.1), the rate of transfer by diffusion is proportional to the concentration gradient and to the area of interface over which the diffusion is occurring. This question has been discussed in Chapter 7 of Volume 1, but some of the important features will be given here.

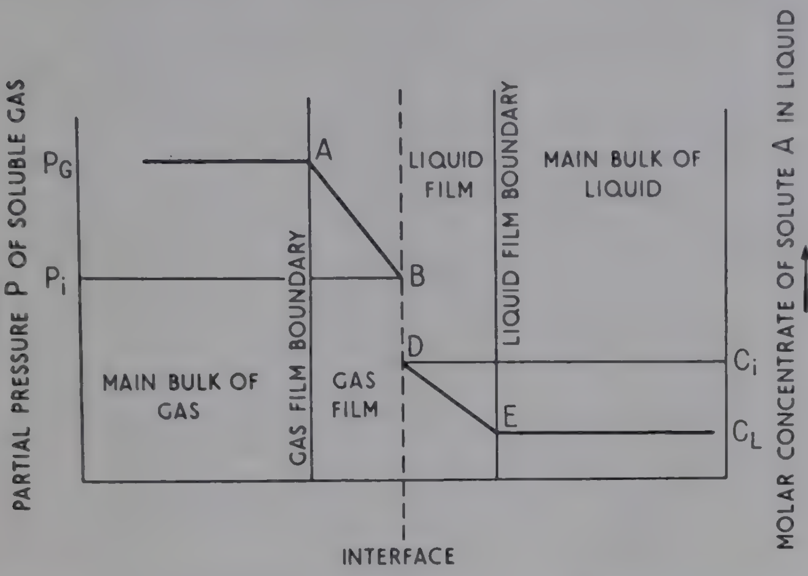


Fig. 19.1. Concentration profile for absorbed component *A*

The direction of transfer of material across the interface is, however, not dependent on the concentration difference, but on the equilibrium relationship. Thus, for a mixture of ammonia or hydrogen chloride and air which is in equilibrium with an aqueous solution, the concentration in the water is many times greater than that in the air. There is, therefore, a very big concentration gradient across the interface: but this is not the controlling factor in the mass transfer, as it is generally assumed that there is no resistance at the interface itself, where equilibrium conditions will exist. The controlling factor will be the rate of diffusion through the two films where all the resistance is considered to lie. The change in concentration of a component through the gas and liquid phases is illustrated in Fig. 19.1. $P_G$ represents the partial pressure in the bulk of the gas phase and $P_i$ the partial pressure at the interface. $C_L$ is the concentration in the bulk of the liquid phase and $C_i$ the concentration at the interface. Thus, according to the above theory, the concentrations at the interface are in equilibrium, and the resistance to transfer is centred in the thin films on either side. We have met this kind of problem in heat transfer across a tube, where we found that the main resistance to transfer lay in the thin films on either side of the wall; here the transfer was by conduction.

### Diffusion of a Gas through a Stagnant Gas

The process of absorption may be regarded as the diffusion of a soluble gas $A$ into a liquid. The molecules of $A$ have to diffuse through a stagnant gas film and then through a stagnant liquid film before entering the main bulk of liquid. If we consider the absorption of a gas consisting of a soluble component $A$ and an insoluble component $B$, we have: (see equation 7.1).

Rate of diffusion of $A$ in direction towards the interface is

$$N_A = -D_V \cdot \frac{\mathrm{d}C_A}{\mathrm{d}z} \qquad \ldots.(19.2)$$

where $N_A$ is the rate of diffusion in mols/unit time-unit area,
$C_A$ is the concentration in mols/unit volume,
$D_V$ is the vapour-phase diffusivity, and
$z$ is the distance in the direction of diffusion.

Rate of diffusion of $B = N_B = -D_V \cdot \frac{\mathrm{d}C_B}{\mathrm{d}z}$ $\qquad \ldots.(19.3)$

But since

$$C_A + C_B = \text{const} = C_T \text{ (say)}, \qquad \ldots.(19.4)$$

$$\frac{\mathrm{d}C_A}{\mathrm{d}z} = -\frac{\mathrm{d}C_B}{\mathrm{d}z}. \qquad \ldots.(19.5)$$

$\therefore$

$$N_B = D_V \cdot \frac{\mathrm{d}C_A}{\mathrm{d}z}. \qquad \ldots.(19.6)$$

Any movement of molecules of $B$ by diffusion away from the interface would lead to a decrease in concentration of $B$ at the interface, since $B$ is not liberated there. This would result in a total concentration difference being set up tending to move both $B$ and $A$ towards the interface. This flow is called the bulk flow and for $B$ will amount to $-D_V \frac{\mathrm{d}C_A}{\mathrm{d}z}$, thus neutralising the flow by diffusion.

The bulk flow of $A$ will be $= -D_V \cdot \frac{\mathrm{d}C_A}{\mathrm{d}z} \cdot \frac{C_A}{C_B}$ $\qquad \ldots.(19.7)$

Hence the rate of absorption of $A$

$$N_A' = \text{flow by diffusion} + \text{bulk flow}$$

$$= -D_V \cdot \frac{\mathrm{d}C_A}{\mathrm{d}z}\left[1 + \frac{C_A}{C_B}\right] \qquad \ldots.(19.8)$$

$\therefore$

$$N_A' = -D_V \cdot \frac{C_T}{C_B} \cdot \frac{\mathrm{d}C_A}{\mathrm{d}z}. \qquad \ldots.(19.9)$$

Integrating over the whole thickness $z_G$ of the film, and representing concentrations at each side of the film by suffixes 1 and 2,

$$N_A' = \frac{D_V \,.\, C_T}{z_G} \,.\, \ln \frac{C_{B2}}{C_{B1}} \quad \ldots.(19.10)$$

$$= \frac{D_V \,.\, P}{\mathbf{R} \,.\, T \,.\, z_G} \,.\, \ln \frac{P_{B2}}{P_{B1}} \text{ (for an ideal gas)} \quad \ldots.(19.11)$$

since $C_T = \dfrac{P}{\mathbf{R}T}$, where **R** is the gas constant, $T$ the absolute temperature, and $P$ the total pressure.

If we write $P_{Bm}$ as the log mean of the partial pressures $P_{B1}$ and $P_{B2}$, then

$$P_{Bm} = \frac{P_{B2} - P_{B1}}{\ln (P_{B2}/P_{B1})} \quad \ldots.(19.12)$$

$$\therefore \qquad N_A' = \frac{D_V P}{\mathbf{R} \,.\, T \,.\, z_G} \,.\, \frac{P_{B2} - P_{B1}}{P_{Bm}}$$

$$= \frac{D_V \,.\, P}{\mathbf{R} \,.\, T \,.\, z_G} \left[ \frac{P_{A1} - P_{A2}}{P_{Bm}} \right] \quad \ldots.(19.13)$$

Hence the rate of absorption of $A$ per unit time over unit area

$$N_A' = k_G' \,.\, P \,.\, \left[ \frac{P_{A1} - P_{A2}}{P_{Bm}} \right] \quad \ldots.(19.14)$$

or

$$N_A' = k_G \left[ P_{A1} - P_{A2} \right] \quad \ldots.(19.15)$$

where

$$k_G' = \frac{D_V}{\mathbf{R} \,.\, T \,.\, z_G}, \text{ and } k_G = \frac{D_V \,.\, P}{\mathbf{R} \,.\, T \,.\, z_G \,.\, P_{Bm}} = \frac{k_G' P}{P_{Bm}} \quad \ldots.(19.16)$$

In the great majority of industrial processes the film thickness is not known, so that the rate equation of immediate use is 19.15 using $k_G$. $k_G$ is known as the gas film transfer coefficient for absorption and is a direct measure of the rate of absorption per unit area of interface with a driving force of unit partial pressure difference.

## Diffusion in the Liquid Phase

The rate of diffusion in liquids is much slower than in gases, and mixtures of liquids may take a long time to reach equilibrium unless agitated. This is partly due to the much closer spacing of the molecules, as a result of which the molecular attractions are more important.

Whilst there is at present no theoretical basis for the rate of diffusion in liquids comparable with the kinetic theory for gases, the basic equation is taken as similar to that for gases, i.e.

$$N_A = -D_L \,.\, \frac{\mathrm{d}C_A}{\mathrm{d}z} \quad \ldots.(19.17)$$

Allowing for bulk flow and integrating,

$$N_A' = -D_L \cdot \left[\frac{C_{A2} - C_{A1}}{z_L}\right] \cdot \frac{C_T}{C_{Bm}}. \quad \ldots.(19.18)$$

where $C_A$, $C_B$ are the molar concentrations of $A$ and $B$,
$z_L$ is the thickness of liquid film through which diffusion occurs,
$C_T$ is the total molar concentration, assumed constant, and
$D_L$ is the diffusivity in the liquid phase.

Since the film thickness is rarely known, equation 19.18 is rewritten as

$$N_A' = k_L\,[C_{A1} - C_{A2}] \quad \ldots.(19.19)$$

which is similar to equation 19.15 for gases.

Here $k_L$ is the liquid film transfer coefficient, which is usually expressed in lb-mols/hr-ft.$^2$ (lb-mols/ft$^3$)

For dilute concentrations, $k_L = \dfrac{D_L}{z_L}$.

**Rate of Absorption based on Two-Film Theory**

In a steady state process of absorption, the rate of transfer of material through the gas film will be the same as that through the liquid film, and we may write the general equation for mass transfer as

$$N_A' = k_G[P_G - P_i] = k_L[C_i - C_L] \quad \ldots.(19.20)$$

where $P_G$ is the partial pressure in the bulk of the gas, $C_L$ is the concentration in the bulk of the liquid, and $P_i$ and $C_i$ are the values of concentration at the interface where equilibrium conditions are assumed to exist.

$$\therefore \quad \frac{k_G}{k_L} = \frac{C_i - C_L}{P_G - P_i} \quad \ldots.(19.21)$$

These conditions may be illustrated geometrically as in Fig. 19.2, where $A.B.F$ is the equilibrium curve.

Point $D$ $(C_L, P_G)$ represents conditions in the bulk of the gas and liquid,
$P_G$ is the partial pressure of $A$ in the main bulk of the gas stream, and
$C_L$ is the average concentration of $A$ in the main bulk of the liquid stream.

Point $A$ $(C_e, P_G)$ represents a concentration $C_e$ in the liquid in equilibrium with $P_G$ in the gas.

Point $B$ $(C_i, P_i)$ represents the concentration $C_i$ in the liquid in equilibrium with $P_i$ in the gas, and gives conditions at the interface.

Point $F$ $(C_L, P_e)$ represents a partial pressure $P_e$ in the gas phase in equilibrium with $C_L$ in the liquid.

Then the driving force causing transfer in the gas phase

$$(P_G - P_i) \equiv DE$$

and the driving force causing transfer in the liquid phase

$$(C_i - C_L) \equiv BE$$

Then $$\frac{P_G - P_i}{C_i - C_L} = \frac{k_L}{k_G}$$

and the concentrations at the interface (point $B$) are found by drawing a line through $D$ of slope $-\frac{k_L}{k_G}$ to cut the equilibrium curve in $B$.

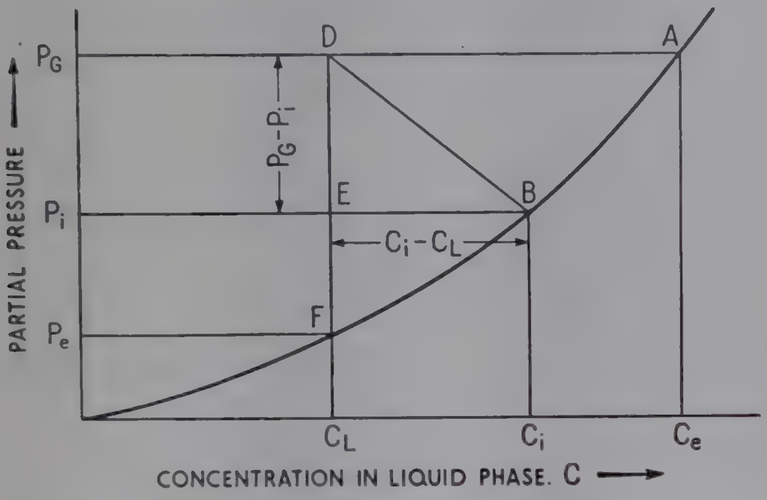


Fig. 19.2. Driving forces in the gas and liquid phases

***Overall Coefficients.*** To obtain a direct measurement of the values of $k_L$ and $k_G$ requires the measurement of the concentration at the interface. These values can only be obtained in very special circumstances, and it has been found of considerable value to use two overall coefficients $K_G$ and $K_L$ defined by the following equation:

$$N_A' = K_G[P_G - P_e] = K_L[C_e - C_L] \qquad \ldots.(19.22)$$

$K_G$ and $K_L$ are known as the overall gas and liquid phase coefficients, respectively.

***Relation between Film and Overall Coefficients.*** The rate of transfer of $A$ can now be written as

$$N_A' = k_G[P_G - P_i] = k_L[C_i - C_L] = K_G[P_G - P_e] = K_L[C_e - C_L] \quad \ldots.(19.23)$$

$$\therefore \qquad \frac{1}{K_G} = \frac{1}{k_G}\left[\frac{P_G - P_e}{P_G - P_i}\right]$$

$$= \frac{1}{k_G}\left[\frac{P_G - P_i}{P_G - P_i}\right] + \frac{1}{k_G}\left[\frac{P_i - P_e}{P_G - P_i}\right]$$

But from above

$$\frac{1}{k_G}=\frac{1}{k_L}\left[\frac{P_G-P_i}{C_i-C_L}\right]$$

$$\therefore \qquad \frac{1}{K_G}=\frac{1}{k_G}+\frac{1}{k_L}\left[\frac{P_G-P_i}{C_i-C_L}\right]\left[\frac{P_i-P_e}{P_G-P_i}\right]$$

$$=\frac{1}{k_G}+\frac{1}{k_L}\left[\frac{P_i-P_e}{C_i-C_L}\right]$$

But $\frac{P_i-P_e}{C_i-C_L}$ is the average slope of the equilibrium curve and, when the solution obeys Henry's Law, we have $\mathscr{H}=\frac{\mathrm{d}P}{\mathrm{d}C}\approx\frac{P_i-P_e}{C_i-C_L}$

$$\therefore \qquad \frac{1}{K_G}=\frac{1}{k_G}+\frac{\mathscr{H}}{k_L} \qquad \ldots.(19.24)$$

Similarly

$$\frac{1}{K_L}=\frac{1}{k_L}+\frac{1}{\mathscr{H}.k_G} \qquad \ldots.(19.25)$$

and

$$\frac{1}{K_G}=\frac{\mathscr{H}}{K_L} \qquad \ldots.(19.26)$$

***Rates of Absorption in Terms of Mol Fractions.***

We can write $\qquad N_A'=k_G''(y-y_i)=K_G''(y-y_e) \qquad \ldots.(19.27a)$

and $\qquad N_A'=k_L''(x_i-x)=K_L''(x_e-x) \qquad \ldots.(19.27b)$

where $x$, $y$ are the mol fractions of the soluble component $A$ in the liquid and gas phases.

$k_G''$, $k_L''$, $K_G''$, $K_L''$ are transfer coefficients defined in terms of mol fractions by equation 19.27*a* and *b*.

If $m$ is the slope of equilibrium curve $\left(\text{approximately } \frac{y_i-y_e}{x_i-x}\right)$, it can then be shown that

$$\frac{1}{K_G''}=\frac{1}{k_G''}+\frac{m}{k_L''} \qquad \ldots.(19.28)$$

which is the same relation as that used for distillation (18.45). It should be noted that equations 19.27*a*, *b* are derived from equations 19.20 and 19.22, and that the total concentration in the liquid phase is assumed to remain constant.

***Factors Influencing the Transfer Coefficient.*** The general influence of the solubility of the gas on the shape of the equilibrium curve, and the resulting influence on film and overall coefficients, will be seen by considering very soluble, almost insoluble, and moderately soluble gases.

(*a*) *Very soluble gas.* Here the equilibrium curve lies close to the $C$-axis and the point $E$ (Fig. 19.2) approaches very close to point $F$. The driving force over the gas film ($DE$) is then approximately equal to the overall driving force ($DF$), so that $k_G$ is approximately equal to $K_G$.

($b$) *Almost insoluble gas.* Here the equilibrium curve rises very steeply so that the driving force $(C_i - C_L)$ ($EB$) in the liquid film becomes approximately equal to the overall driving force $(C_e - C_L)$ ($AD$). In this case $k_L$ will be approximately equal to $K_L$.

($c$) *Moderately soluble gas.* Here both films offer an appreciable resistance, and the point $B$ at the interface must be located by drawing a line through $D$ of slope $-\dfrac{k_L}{k_G} = -\dfrac{P_G - P_i}{C_i - C_L}$.

In most experimental work, the concentration at the interface cannot be measured directly, and only the overall coefficients will therefore be found. To obtain values for the film coefficients, we must utilise the relations between $k_G$, $k_L$ and $K_G$ as discussed on page 706.

## GAS ABSORPTION EQUIPMENT

From the examinations given above of the diffusional nature of absorption, we can see that one of the outstanding requirements is to provide as large an interfacial area of contact as possible between the phases. For this purpose, columns similar to those used for distillation are suitable. In addition, equipment in which the gas is blown into a liquid which is agitated by some stirrer, and a few special forms of units have been used.

### Plate Columns

These are similar in construction to those described in Chapter 18, and may be fitted either with perforated plates or with bubble cap trays. Since the ratio of liquor rate to gas rate is greater than with distillation, the slot area will be rather less and the downcomers rather larger. On the whole, plate efficiencies have been found to be less than with the distillation equipment, and to range from 20 to 80%.

The plate column is a common type of equipment for large installations, but when the diameter of the column is less than 6 feet, packed columns are more often used; for the handling of very corrosive fluids, packed columns are frequently preferred for larger units.

### Packed Columns

The main parts of an installation, as indicated in Fig. 19.3, are (1) the column, (2) arrangements for the liquor and gas inlet and outlet, (3) the packing, (4) the gas blower.

1. *The Column.*

When handling corrosive gases such as chlorine, hydrogen fluoride, and oxides of nitrogen, the column is often made of stoneware, or of plastic material such as Keebush. These columns are made of rings with bell and spigot joints if stoneware, or with flange joints if plastic, and are suitable for diameters up to about 4 feet. Stainless steel is being increasingly used for columns which can then be made in one piece and are suitable for high pressure operation. It is sometimes desirable to operate a number of towers in series rather than to build one very tall unit. In tall columns, it is necessary to include a number of redistributing points for the liquor in order to reduce channelling.

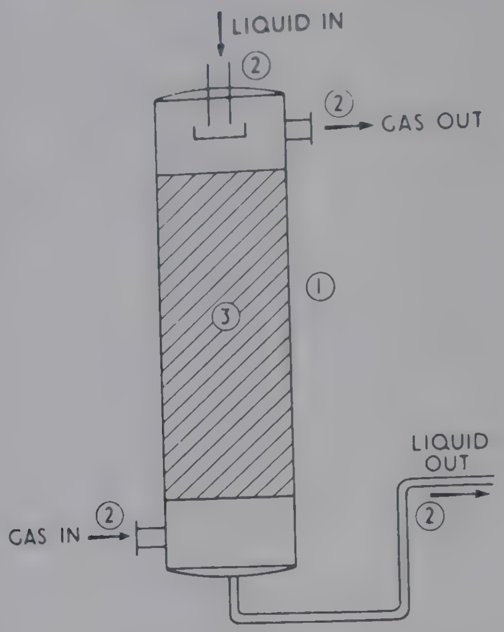


Fig. 19.3. General arrangement of packed column

2. *Inlet and Outlet Arrangements.*

The liquor is usually run into the top of the tower, either through a series of nozzles or over distributor plates. The gas enters below the packing and passes upwards, and a liquid seal is therefore necessary on the liquor outlet pipe to prevent the gas leaving with the liquor. Whilst it is always necessary to pay attention to the method of feeding the liquid, it is particularly important when the liquid rate is insufficient to wet the packing completely. Two arrangements are illustrated in Figs. 19.4 and 19.5 (taken from MORRIS and JACKSON[(3)]).

3. *Packing Materials.*

Some features regarding the selection of packing materials have been given in Chapter 11, but a summary of the main points to be considered is given below:

(*a*) Durability and corrosion resistance.

(*b*) Free space per cubic foot of packed space. This controls the liquor hold-up in the column, as well as the pressure drop across it.

(*c*) Wetted surface area per unit volume of packed space. This is very important since it determines the interfacial surface. It is rarely equal to the actual geometrical surface because the packing is not usually completely wetted by the fluid.

(*d*) Freedom from channelling.

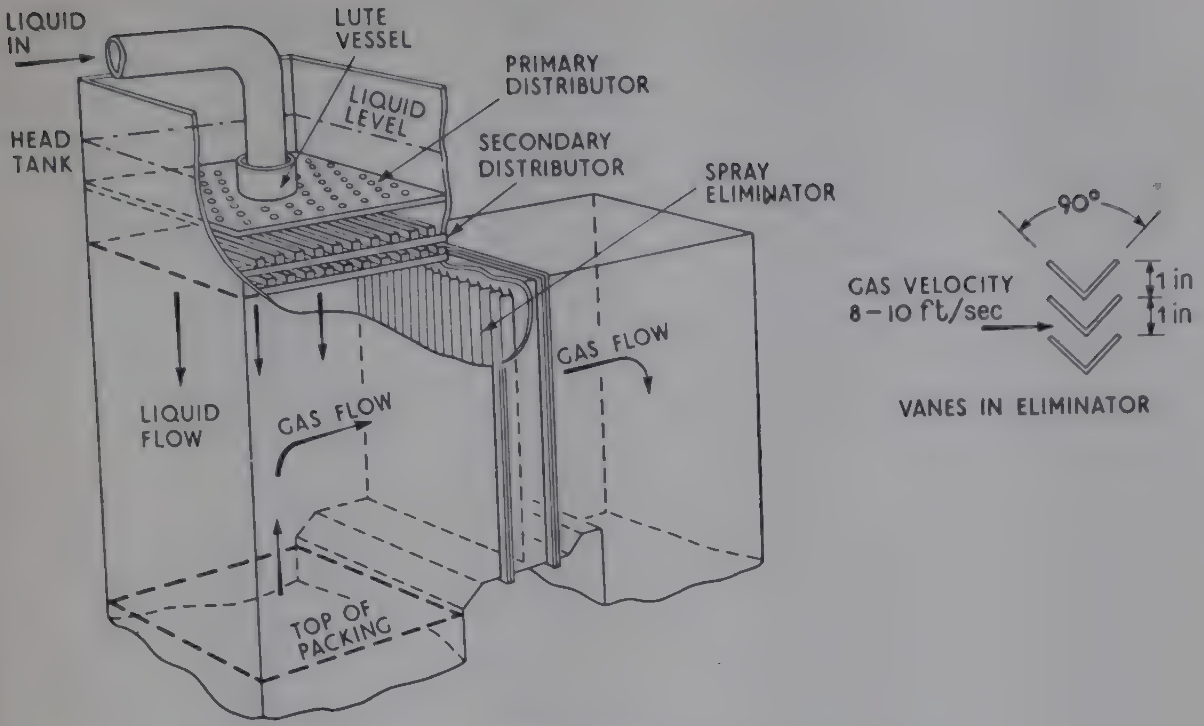

LIQUID IN
LUTE VESSEL
LIQUID LEVEL
PRIMARY DISTRIBUTOR
HEAD TANK
SECONDARY DISTRIBUTOR
SPRAY ELIMINATOR
90°
GAS VELOCITY 8–10 ft/sec
1 in
1 in
VANES IN ELIMINATOR
GAS FLOW
LIQUID FLOW
GAS FLOW
TOP OF PACKING

Fig. 19.4. Splash-type distributor for liquid

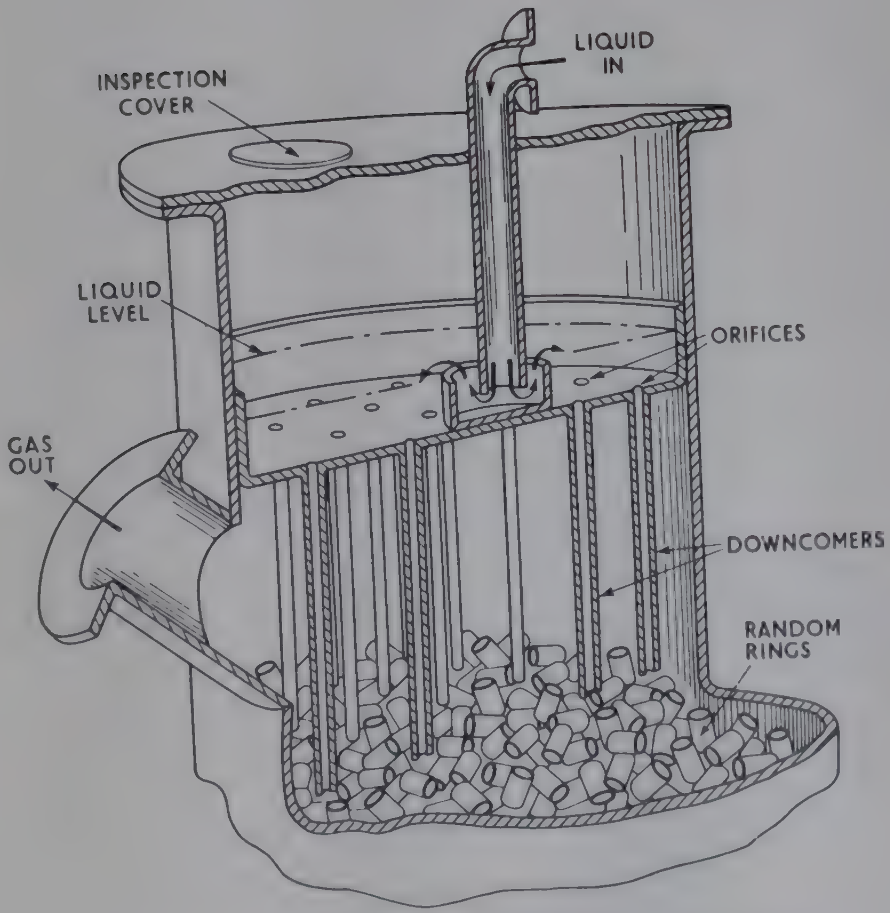

LIQUID IN
INSPECTION COVER
LIQUID LEVEL
ORIFICES
GAS OUT
DOWNCOMERS
RANDOM RINGS

Fig. 19.5. Non-splash type distributor for liquid

(*e*) Frictional resistance to the flow of gas. This affects the pressure drop over the column.

(*f*) Weight per cubic foot of packed space.

(*g*) Stability of the packing in position—in particular, absence of lateral pressure.

(*h*) Cost per square foot of effective surface.

(*i*) Porosity, particularly of ceramic materials, and ease of wetting.

The most commonly used packing materials are rings or saddles, some particulars of which are given in Table 19.2, taken from a paper by WILLIAMSON$^{(28)}$.

4. *Pumping Arrangements.*

In some installations, gas may be received from a previous process under pressure, and in this case there is no need to provide a blower. When this is not the case, a simple blower is commonly used; the various types are described in Chapter 5, Volume I. It must be remembered that simple single stage blowers only develop low pressures, and this may limit the height of the column.

***Liquid and Gas Rates in Packed Towers.*** The drop in pressure for the flow of gas and liquid over packings has been discussed in Chapter 11. It is important to see that, during operation, the tower does not reach flooding conditions. In addition, every effort should be made to have as high a liquid rate as possible, in order to obtain complete wetting of the packing. If the pressure drop must be kept very low, it is usually better to use a plate column.

***Importance of Liquid Rate.*** With low liquid rates, the whole of the surface of the packing is not completely wetted. This can be seen very readily by allowing a coloured liquid to flow over packing contained in a glass tube. From the flow patterns, it is at once obvious how little of the surface is wetted until the rate is quite high. This difficulty of wetting can sometimes be overcome by having considerable recirculation of the liquid over the tower, but in other cases poor wetting will have to be accepted because of the low volume of liquid available. In selecting a packing, it is desirable to choose the form which will give as near complete wetting as possible. The minimum liquid rate at which the packing is fully wetted is known as the minimum irrigation rate (M.I.R.), which has been discussed in Chapter 11.

### Spray Towers

In the spray tower, the gas enters at the bottom and the liquid is introduced through a series of sprays at the top. The performance of these units is generally rather poor, because the droplets tend to coalesce after they have fallen through a few feet, and the interfacial surface is thereby seriously reduced. Although there is considerable turbulence in the gas phase, there is little circulation of the liquid within the drops, and the resistance of the equivalent liquid film

TABLE 19.2. *Packing Data*

| 1<br>Item No. | 2<br>Packing | 3<br>Material | 4<br>Dimensions | 5<br>Number of packing elements per cu. ft of packed volume | 6<br>Height of a gas-film transfer unit. (ft) | 7<br>Pressure drop velocity (heads per ft of packing) | 8<br>Minimum effective liquor rate (lb/hr-sq. ft) (water) | 9<br>Approx. cost of packing installed (shillings/cu. ft) | 10<br>Optimum gas velocity (loading velocities in brackets) (ft/sec) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | Plain grids | Metal | 1″ pitch × 1″ deep × $\frac{1}{16}$″ thick | — | 3·5 | 3–4 | 1300 | 60 | 10 (11) |
| 2 | | Timber | 1″ pitch × 1″ deep × $\frac{1}{4}$″ thick | — | 2·9 | 8–10 | 1500 | 12 | 5–7 (9) |
| 3 | Serrated grids | Timber | 2″ pitch × 2″ deep × $\frac{3}{8}$″ thick | — | 5·9 | 3 | 660 | 9 | 7–10 (12) |
| 4 | Stacked rings | Ceramic | 3″ o.d. × 2″ deep × $\frac{3}{8}$″ thick | 65 | 3·7 | 12–20 | 1300 | 9 | 4–5 (5) |
| 5 | | Ceramic | 2″ o.d. × 2″ deep × $\frac{1}{4}$″ thick | 210 | 2·4 | 40–60 | 2000 | 22 | 3 (3·5) |
| 6 | Random rings | Ceramic | 3″ o.d. × 3″ deep × $\frac{3}{8}$″ thick | 52 | 2·6 | 50–80 | 1050 | 5 | 2·5–3·5 (5·5) |
| 7 | | Metal | 2″ o.d. × 2″ deep × $\frac{1}{16}$″ thick | 175 | 1·5 | 80–200 | 1500 | 17 | 2·3 (3·5) |
| 8 | | Ceramic | 2″ o.d. × 2″ deep × $\frac{1}{4}$″ thick | 165 | 1·6 | 150–400 | 1500 | 9 | 1·5–2 (3) |
| 9 | Solid packing | Coke | 1″–2″ | — | 0·8 | 500–800 | 1700 | 3 | 1–1·5 (2) |

*Notes* (1) The optimum gas velocity is based on air at atmospheric conditions. When the gas density is not equal to that of atmospheric air, the optimum velocities given in Column 10 must be multiplied by the ratio $\left\{\frac{\text{Density of atmospheric air}}{\text{Actual density of gas}}\right\}^{\frac{1}{2}}$. The figures in brackets are the loading rates for air at the corresponding minimum effective irrigation rates (M.I.R.). Above the loading rate, pressure drops rise steeply with increased gas rates. Loading rates are roughly 60% of the flooding rates for random packings and 35% for stacked packings.

(2) The velocity heads are based on "empty tower" velocities. The lower figures hold for wetting rates which correspond to the M.I.R., and the upper for irrigation rates 6 times the M.I.R.

(3) The height of a liquid-film transfer unit $H_L$ is 2 ft for all packings except the 1 in. grids, for which it is 1·5 ft.

(4) The height of a gas-film transfer unit was determined by cooling water with air.

(5) "Ceramic" refers to an inexpensive ceramic material based on ferruginous shale.

tends to be high. Spray towers are therefore useful only where the main resistance to mass transfer lies within the gas phase, and have consequently been used with moderate success for the absorption of ammonia in water. They are also used as air humidifiers; in this case the whole of the resistance lies within the gas phase.

## CAPACITY OF PACKED TOWERS

Fig. 19.6 illustrates the conditions that occur during the steady operation of a countercurrent gas-liquid absorption tower. It is convenient to express the concentrations of the streams in terms of mols of solute gas per mol of inert gas in the gas phase, and as mols of solute gas per mol of solute free liquid in the liquid phase. The actual area of interface between the two phases is not known, and the term $a$ is introduced as the interfacial area per unit volume of the column. On this basis the general equation (19.20) for mass transfer can be written as:

$$N_A' \,.\, S \,.\, \mathrm{d}Z \,.\, a = k_G a \,.\, (P_G - P_i)\, S\, \mathrm{d}Z$$
$$= k_L a \,.\, (C_i - C_L)\, S\, \mathrm{d}Z \quad \ldots.(19.29)$$

where $N_A'$ = mols of solute absorbed per unit time and unit interfacial area,
$a$ = surface area of interface per unit volume of column,
$S$ = cross sectional area of column, and
$Z$ = height of packed section,

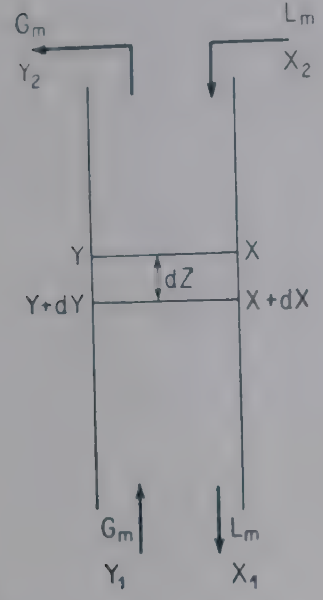


Fig. 19.6. Countercurrent absorption tower

The interfacial area for transfer $= a \,.\, \mathrm{d}V = a \,.\, S \,.\, \mathrm{d}Z \quad \ldots.(19.30)$

**Height of Column based on Conditions in Gas Film**

Let $G_m$ = mols of inert gas/(unit time) . (unit cross section of tower),
$L_m$ = mols of solute-free liquor/(unit time) (unit cross section of tower),
$Y$ = mols of solute gas $A$/mol of inert gas $B$ in gas phase, and
$X$ = mols of solute $A$/mol of inert solvent in liquid phase.

Consider any plane at which the mol ratios of the diffusing material in the gas and liquid phases are $Y$ and $X$. Then over a small height d$Z$, the mols of gas leaving the gas phase will equal the mols taken up by the liquid. This gives:

$$S \,.\, G_m \,.\, \mathrm{d}Y = S \,.\, L_m\, \mathrm{d}X \quad \ldots.(19.31)$$

But

$$G_m S\, \mathrm{d}Y = N_A'(a \,.\, \mathrm{d}V) = k_G a[P_G - P_i]\, S \,.\, \mathrm{d}Z \quad \ldots.(19.32)$$

Since

$$P_G = \frac{Y}{1+Y} \,.\, P$$

$$G_m \,.\, \mathrm{d}Y = k_G a P \left[\frac{Y}{1+Y} - \frac{Y_i}{1+Y_i}\right] . \,\mathrm{d}Z$$

$$= k_G a P \left[\frac{Y - Y_i}{(1+Y)(1+Y_1)}\right] . \,\mathrm{d}Z$$

Hence the height of column $Z$ required to achieve a change in $Y$ from $Y_1$ at the bottom to $Y_2$ at the top of the column is given by

$$\int_0^Z dZ = Z = \frac{G_m}{k_G a P} \int_{Y_2}^{Y_1} \frac{(1+Y)(1+Y_i)\,dY}{Y - Y_i} \qquad \ldots.(19.33)$$

which for weak mixtures can be written as

$$Z = \frac{G_m}{k_G a P} \int_{Y_2}^{Y_1} \frac{dY}{Y - Y_i} \qquad \ldots.(19.34)$$

In this analysis it has been assumed that $k_G$ is a constant throughout the column, and provided the concentration changes are not too large this will be reasonably true.

### Height of Column based on Conditions in Liquid Film

A similar analysis may be made in terms of the liquid film. Thus from equations 19.29, 19.30 and 19.31, we can write,

$$S\,.\,L_m\,dX = k_L a(C_i - C_L)\,S\,.\,dZ \qquad \ldots.(19.35)$$

where the concentrations $C$ are in terms of mols of solute per unit volume of liquor. If $C_T = \dfrac{\text{mols of solute + solvent}}{\text{volume of liquor}}$,

$$\frac{C}{C_T - C} = \frac{\text{mols of solute}}{\text{mols of solvent}} = X$$

whence

$$C = \frac{X}{1+X} C_T \qquad \ldots.(19.36)$$

The transfer equation (19.35) may now be written

$$L_m\,.\,dX = k_L a C_T \left[\frac{X_i}{1+X_i} - \frac{X}{1+X}\right] dZ$$

$$= k_L a C_T \left[\frac{X_i - X}{(1+X_i)(1+X)}\right] dZ$$

$$\therefore \quad \int_0^Z dZ = Z = \frac{L_m}{k_L a C_T} \cdot \int_{X_2}^{X_1} \frac{(1+X_i)(1+X)\,dX}{X_i - X} \qquad \ldots.(19.37)$$

and for dilute concentrations this gives

$$Z = \frac{L_m}{k_L a C_T} \int_{X_2}^{X_1} \frac{dX}{X_i - X} \qquad \ldots.(19.38)$$

where $C_T$ and $k_L$ have been taken as constant over the column.

### Height based on Overall Coefficients

If the driving force based on the gas concentration is written as $Y - Y_e$ and the overall gas transfer coefficient as $K_G$, then the height of the tower for dilute concentrations becomes

$$Z = \frac{G_m}{K_G a P} \int_{Y_2}^{Y_1} \frac{dY}{Y - Y_e} \qquad \ldots.(19.39)$$

or in terms of the liquor concentration as

$$Z = \frac{L_m}{K_L a C_T} \cdot \int_{X_2}^{X_1} \frac{dX}{X_e - X} \qquad \ldots.(19.40)$$

***Equations for dilute concentrations.*** As the mol fraction is approximately equal to the mol ratio at dilute concentrations, considering the gas film, we can write,

$$Z = \frac{G_m}{K_G a P} \int_{Y_2}^{Y_1} \frac{dY}{Y - Y_e} = \frac{G_m}{K_G a P} \int_{y_2}^{y_1} \frac{dy}{y - y_e} \qquad \ldots.(19.41)$$

and considering the liquid film,

$$Z = \frac{L_m}{K_L a C_T} \int_{X_2}^{X_1} \frac{dX}{X_e - X} = \frac{L_m}{K_L a C_T} \int_{x_2}^{x_1} \frac{dx}{x_e - x} \qquad \ldots.(19.42)$$

### The Operating Line and Graphical Integration for Height of Column

If we take a material balance on the solute from the bottom of the column to any plane where the mol ratios are $Y$ and $X$, we get for unit area of cross-section

$$G_m (Y_1 - Y) = L_m (X_1 - X) \qquad \ldots.(19.43a)$$

or

$$Y_1 - Y = \frac{L_m}{G_m} (X_1 - X) \qquad \ldots.(19.43b)$$

This is the equation of a straight line of slope $L_m/G_m$, which passes through the point $(X_1, Y_1)$. It can be seen by writing a material balance over the whole column that the same line passes through the point $(X_2, Y_2)$. This line is known as the operating line and represents the conditions at any point in the column; it is similar to the operating line used in Chapter 18. Fig. 19.7 illustrates typical conditions for the case of moist air and sulphuric acid or caustic soda solution, where the main resistance lies in the gas phase.

The equilibrium curve is represented by the line $OFR$, and the operating line is given by $AB$, $A$ corresponding to the concentrations at the bottom of the column and $B$ to those at the top of the column.

$D$ represents the condition of the bulk of the liquid and gas at any point in the column, and has co-ordinates $X$ and $Y$. Then, if the gas film is controlling the process, $Y_i$ equals $Y_e$, and is given by a point $F$ on the equilibrium curve, with co-ordinates $X$ and $Y_i$. The driving force causing transfer is then given by the distance $DF$. It is therefore possible to evaluate the expression $\int_{Y_2}^{Y_1} \frac{dY}{Y - Y_i}$ by selecting values of $Y$, reading off from the diagram the corresponding values of $Y_i$, and thus calculating $\frac{1}{Y - Y_i}$.

If the liquid film controls the process, $X_i$ equals $X_e$ and the driving force $X_i - X$ is given in the diagram by the line $DR$. Thus, the evaluation of $\int_{X_2}^{X_1} \frac{dX}{X_i - X}$ may be effected in the same way as for the gas film.

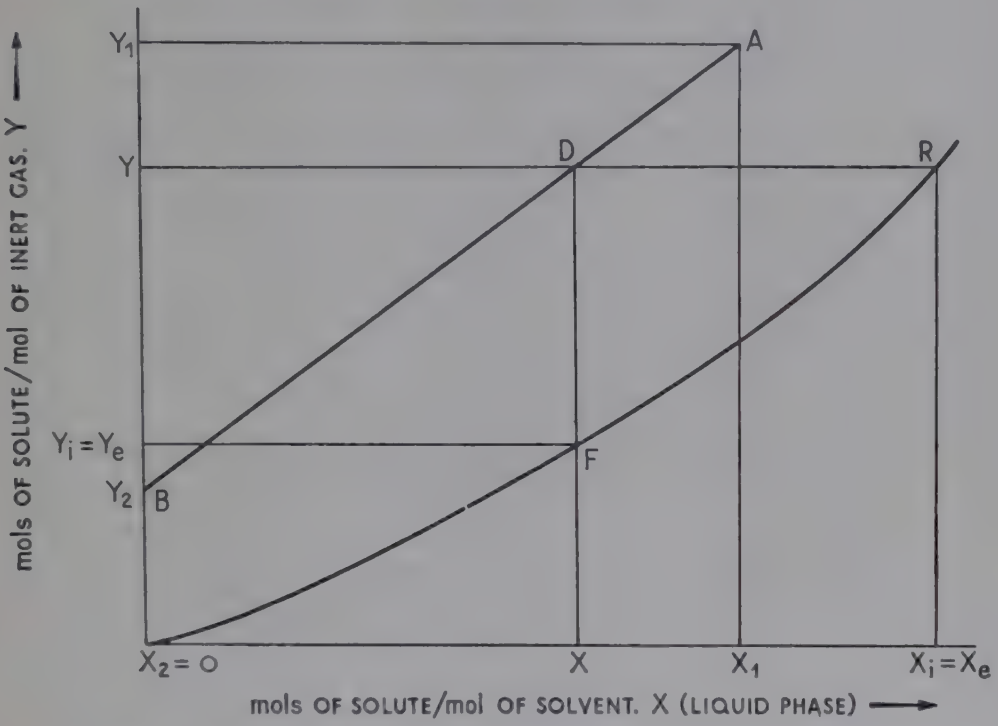

Y₁
A
Y
D
R
Yi = Ye
F
Y2
B
X2 = 0
X
X1
Xi = Xe
mols OF SOLUTE/mol OF INERT GAS. Y
mols OF SOLUTE/mol OF SOLVENT. X (LIQUID PHASE)

Fig. 19.7. Driving force in gas and liquid-film controlled processes

### Special Case when Equilibrium Curve is a Straight Line

If over the range of concentrations considered the equilibrium curve is a straight line, it is permissible to use an average value of the driving force over the column. For dilute concentrations, we can write that, over a small height $dZ$ of column, the absorption is given by

$$N'_A Sa\, dZ = G_m S\, dy = K_G aSP(y - y_e)\, dZ \qquad \ldots.(19.44)$$

If

$$y_e = mx + c \qquad \ldots.(19.45)$$

we have

$$y_{e2} = mx_2 + c$$

and

$$y_{e1} = mx_1 + c$$

so that

$$m = \frac{y_{e2} - y_{e1}}{x_2 - x_1} \qquad \ldots.(19.46)$$

Further, if we take a material balance over the lower portion of the column,

$$L_m(x - x_1) = G_m(y - y_1)$$

and

$$x = x_1 + \frac{G_m}{L_m}(y - y_1) \qquad \ldots.(19.47)$$

From equation 19.44,

$$\int_{y_2}^{y_1} \frac{dy}{y - y_e} = \int_0^Z \frac{K_G a P}{G_m} dZ \qquad \ldots.(19.48)$$

$$= \int_{y_2}^{y_1} \frac{dy}{y - m\left[x_1 + \frac{G_m}{L_m}(y - y_1)\right] - c}$$

(from equations 19.45 and 19.47)

$$= \frac{1}{1 - \frac{mG_m}{L_m}} \ln \frac{y_1 - mx_1 - c}{y_2 - m\left[x_1 + \frac{G_m}{L_m}(y_2 - y_1)\right] - c}$$

$$= \frac{1}{1 - \frac{y_{e1} - y_{e2}}{x_1 - x_2} \cdot \frac{x_1 - x_2}{y_1 - y_2}} \ln \frac{(y - y_e)_1}{(y - y_e)_2}$$

(from equations 19.45, 19.46 and 19.47)

$$= \frac{y_1 - y_2}{(y - y_e)_1 - (y - y_e)_2} \ln \frac{(y - y_e)_1}{(y - y_e)_2}$$

$$= \frac{y_1 - y_2}{(y - y_e)_{\text{lm}}}$$

where $(y - y_e)_{\text{lm}}$ is the logarithmic mean value of $y - y_e$.

Substituting in equation 19.48,

$$\frac{K_G a P}{G_m} Z = \frac{y_1 - y_2}{(y - y_e)_{\text{lm}}}$$

$$\therefore \qquad aS \,.\, Z \,.\, N_A' = G_m(y_1 - y_2)S = K_G aSP(y - y_e)_{\text{lm}} Z \qquad \ldots.(19.49)$$

and in terms of mol ratios,

$$aS \,.\, Z \,.\, N_A' = G_m(Y_1 - Y_2)S = K_G aSP(Y - Y_e)_{\text{lm}} Z \qquad \ldots.(19.50)$$

Thus, the logarithmic mean of the driving forces at the top and the bottom of the column may be used.

For concentrated solutions, we have,

$$aS \, . \, Z \, . \, N_A' = G_m(Y_1 - Y_2)S = K_G aS\phi P(Y - Y_e)_{lm}Z \quad ....(19.51)$$

It is necessary to introduce the factor $\phi$ since $Y$ is not directly proportional to $P$. The value of $\phi$ may be found from the relation,

$$\phi Y = \frac{Y}{1 + Y} \quad ....(19.52)$$

whence $\phi = \frac{1}{1 + Y}$. Although the value of $\phi$ will change slightly over the column, an average value will generally be sufficiently accurate.

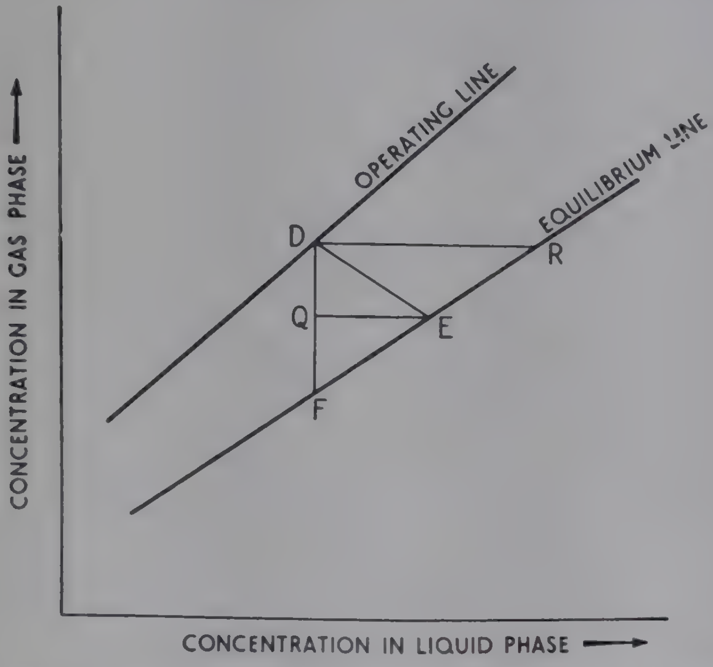


Fig. 19.8. Driving force when equilibrium curve is a straight line

It is of interest to note from Fig. 19.8 that, as long as the ratio $k_L/k_G$ remains constant (i.e. if the slope of $DE$ is constant), then the ratio of $DQ$ the driving force through the gas phase divided $DF$ the driving force assuming all the resistance to be in the gas phase will be a constant. Thus, the use of the driving force $DF$ is satisfactory even if the resistance does not lie wholly in the gas phase. The coefficient $k_G$ on this basis is not an accurate value for the gas film coefficient, but is proportional to it. It follows that, if the equilibrium curve is straight, either the gas film or the liquid film coefficient may be used. This simplification is of considerable value.

### Capacity of Tower in Terms of Partial Pressures for High Concentrations

A material balance taken between the bottom of the column and some plane where the partial pressure in the gas phase is $P_G$ and the concentration in the liquid is $X$ will give:

$$L_m(X_1 - X) = G_m\left[\frac{P_{G1}}{P - P_{G1}} - \frac{P_G}{P - P_G}\right] \quad ....(19.53)$$

Over a small height of the column d$Z$, we then have

$$L_m\,\mathrm{d}X = \frac{G_m P}{(P - P_G)^2}\,\mathrm{d}P_G = k_G a(P_G - P_i)\,\mathrm{d}Z \qquad \ldots.(19.54)$$

$$= \frac{k_G P_{Bm}}{P}\,.\,a\,.\,P\,\frac{(P_G - P_i)}{P_{Bm}}\,\mathrm{d}Z$$

$$= k_G{}'a\,.\,P\,\frac{(P_G - P_i)}{P_{Bm}}\,\mathrm{d}Z$$

$$\therefore \quad \int_0^Z \mathrm{d}Z = G_m \int_{P_{G2}}^{P_{G1}} \frac{P_{Bm}\,\mathrm{d}P_G}{k_G{}'a(P - P_G)^2(P_G - P_i)} \qquad \ldots.(19.55)$$

The advantage of using $k_G{}'$ instead of $k_G$ is that $k_G{}'$ is independent of concentration, but this equation is almost unmanageable in practice. If a substantial amount of the gas is absorbed from a concentrated mixture, $k_G{}'$ will still change as a result of a reduced gas velocity, although it is independent of concentration.

## The Transfer Unit

The group $\int \frac{\mathrm{d}y}{y - y_e}$, which we have met before in Chapter 18, has been defined by CHILTON and COLBURN$^{(14)}$ as the number of overall gas transfer units $\mathbf{N}_{OG}$. The application of this group to the countercurrent conditions in the absorption tower is given below.

Over a small height d$Z$, the partial pressure of the diffusing component $A$ will change by an amount d$P_G$. Then the mols of $A$ transferred are given by

$$\text{(change in mol fraction)} \times \text{(total mols of gas)} \qquad \ldots.(19.56)$$

$$\therefore \quad K_G a(P_G - P_e)\,\mathrm{d}Z = \frac{\mathrm{d}P_G}{P}\,G_m \qquad \ldots.(19.57)$$

(for dilute concentrations)

$$\therefore \quad \int_{P_{G2}}^{P_{G1}} \frac{\mathrm{d}P_G}{P_G - P_e} = \int_0^Z \frac{K_G a P}{G_m}\,\mathrm{d}Z \qquad \ldots.(19.58)$$

or in terms of mol fractions

$$\int_{y_2}^{y_1} \frac{\mathrm{d}y}{y - y_e} = \int_0^Z K_G a\,\frac{P}{G_m}\,\mathrm{d}Z = K_G a\,\frac{P}{G_m}\,Z. \qquad \ldots.(19.59)$$

The number of overall gas transfer units $\mathbf{N}_{OG}$ is an integrated value of the change in composition per unit driving force, and therefore represents the difficulty of the separation. As in Chapter 18 (page 652) we can write equation 19.59 as

$$\mathbf{N}_{OG} = \frac{\text{Height of column}}{\text{Height of transfer unit}} = \frac{Z}{\mathbf{H}_{OG}} \qquad \ldots.(19.60)$$

Then the height of the overall gas transfer unit $\mathbf{H}_{OG} = \dfrac{G_m}{PK_G a}$ ....(19.61)

If the driving force is taken over the gas film only, we obtain the height of gas film transfer unit $\mathbf{H}_G = \frac{G_m}{Pk_Ga}$. Similarly for the liquid film, the height of the overall liquid transfer unit $= \frac{L_m}{K_L a C_T}$. ....(19.62)

The height of the liquid film transfer unit, $\mathbf{H}_L = \frac{L_m}{k_L a C_T}$ ....(19.63)

where $C_T$ is the mean molar density of the liquid.

In this analysis, it has been assumed that the total number of mols of gas and liquid remain the same. This is true in absorption only when a small change in concentration takes place. With distillation, the total number of mols of gas and liquid does remain approximately constant so that no difficulty arises in that case.

*Concentrated Solutions.* With concentrated solutions, we must allow for the change in the total number of mols flowing, because the molar flow will decrease up the column if the amount of absorption is large.

We must then use the following equation in place of equation 19.57,

$$aS\,\mathrm{d}Z\,.\,N_A' = S\,\mathrm{d}\left[\frac{P_G}{P}G_m\right] \qquad \text{....(19.64)}$$

COLBURN(17) has shown that, under these conditions, the number of transfer units is given by

$$\mathbf{N}_{OG} = \int_{y_2}^{y_1} \frac{\mathrm{d}y}{y - y_e}\,\frac{(1-y)_{\mathrm{lm}}}{1-y} \qquad \text{....(19.65)}$$

where $(1 - y)_{\mathrm{lm}}$ is the logarithmic mean of $1 - y$ and $1 - y_i$.

### The Importance of Liquid and Gas Flow Rates and the Slope of the Equilibrium Curve

For a packed tower operating with dilute concentrations we have, since $x \simeq X_1$ and $y \simeq Y_1$,

$$G_m(y_1 - y_2) = L_m(x_1 - x_2) \qquad \text{....(19.66)}$$

where, as before, $x$ and $y$ are the mol fractions of solute in the liquid and gas phases, and

$G_m$ and $L_m$ are the gas and liquid molar flow rates per unit area on a solute free basis.

A material balance between the top and some plane where the mol fractions are $x$, $y$ gives

$$G_m(y - y_2) = L_m(x - x_2) \qquad \text{....(19.67)}$$

If the entering solvent is free from solute, then $x_2 = 0$ and

$$x = \frac{G_m}{L_m}(y - y_2) \tag{19.67a}$$

But the number of overall transfer units is given by

$$\mathbf{N}_{OG} = \int_{y_2}^{y_1} \frac{dy}{y - y_e}$$

For dilute concentrations, Henry's law holds and $y_e = mx$.

$$\therefore \quad \mathbf{N}_{OG} = \int_{y_2}^{y_1} \frac{dy}{y - \dfrac{mG_m}{L_m}(y - y_2)}$$

$$= \int_{y_2}^{y_1} \frac{dy}{y\left[1 - \dfrac{mG_m}{L_m}\right] + \dfrac{mG_m}{L_m} y_2}$$

$$\therefore \quad \mathbf{N}_{OG} = \frac{1}{1 - \dfrac{mG_m}{L_m}} \ln\left[\left(1 - \frac{mG_m}{L_m}\right)\frac{y_1}{y_2} + \frac{mG_m}{L_m}\right] \quad \ldots\ldots (19.68)$$

Colburn[17] has shown that this equation can usefully be plotted as shown in Fig. 19.9 which is taken from his paper. In this plot the number of transfer units $\mathbf{N}_{OG}$ is shown for values of $y_1/y_2$ using $\dfrac{mG_m}{L_m}$ as a parameter. We can now see that the greater is $\dfrac{mG_m}{L_m}$, the greater will be the value of $\mathbf{N}_{OG}$ for a given ratio of $\dfrac{y_1}{y_2}$.

From equation (19.67a),

$$\frac{L_m}{G_m} = \frac{y_1 - y_2}{x_1} = \frac{y_1 - y_2}{y_{e1}/m}$$

$$\therefore \quad \frac{mG_m}{L_m} = \frac{y_{e1}}{y_1 - y_2}$$

where $y_{e1}$ is value of $y$ in equilibrium with $x_1$.

On this basis, the lower the value of $\dfrac{mG_m}{L_m}$, the lower will be $y_{e1}$, and hence the weaker the exit liquor. Colburn has suggested that the economic range for $\dfrac{mG_m}{L_m}$ lies from 0·5 to 0·8. If the value of $\mathbf{H}_{OG}$ is known, the quickest way of obtaining a good indication of the required height of the column is by using Fig. 19.9.

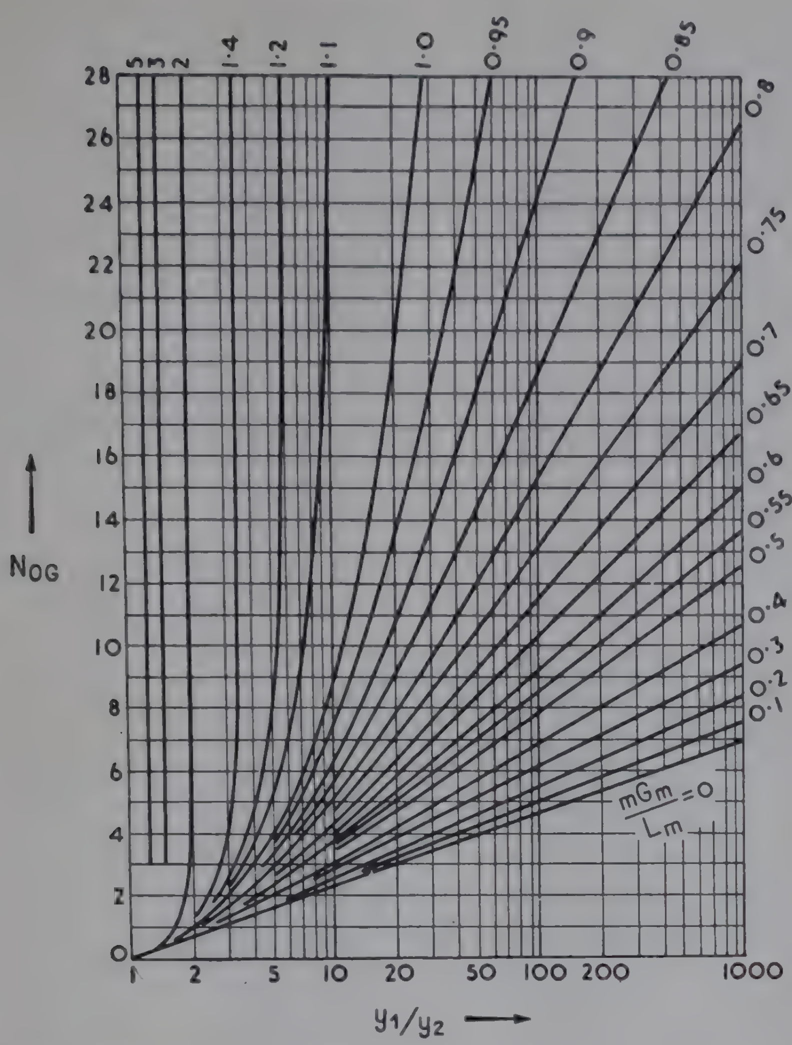


Fig. 19.9. Number of transfer units $N_{OG}$ as a function of $y_1/y_2$, with $\dfrac{mG_m}{L_m}$ as parameter

**Example.** Gas, from a petroleum distillation column, has its concentration of $H_2S$ reduced from 0·03 mol $H_2S$ per mol of inert hydrocarbon gas, to $\dfrac{1}{100}$ of this value, by scrubbing with a triethanolamine-water solvent in a countercurrent tower, operating at 27°C and at atmospheric pressure. The equilibrium relation for the solution may be taken as $Y_e = 2X$.

The solvent enters the tower free of $H_2S$ and leaves containing 0·013 mols of $H_2S$ per mol of solvent. If the flow of inert gas is 10 lb-mol/hr-sq. ft of tower cross-section, calculate

(*a*) the height of the absorber necessary, and

(*b*) the number of transfer units $N_{OG}$ required.

The overall coefficient for absorption $K_G''a$ may be taken as 8 lb-mol/hr-cu. ft-unit mol fraction driving force.

**Solution**

Driving force at top of column $= Y_2 - Y_{2e} = 0{\cdot}0003$

Driving force at bottom of column $= Y_1 - Y_{1e} = 0{\cdot}03 - 0{\cdot}026 = 0{\cdot}004$

Logarithmic mean driving force $= \dfrac{0{\cdot}004 - 0{\cdot}0003}{\ln \dfrac{0.004}{0.0003}}$

$$= \frac{0{\cdot}0037}{2{\cdot}59} = 0{\cdot}00143$$

From equation 19.49,
$$G_m(Y_1 - Y_2)S = K_G a P(Y - Y_e)_{lm} \, . \, S \, . \, Z.$$

i.e.
$$G_m(Y_1 - Y_2) = K_G'' a(Y - Y_e)_{lm} \, . \, Z$$

$\therefore$
$$10(0{\cdot}03 - 0{\cdot}0003) = 8 \times 0{\cdot}00143Z$$

$\therefore$
$$Z = \frac{0{\cdot}297}{0{\cdot}01144} = 25{\cdot}9 = \underline{\underline{26 \text{ ft}}} \text{ (say)}$$

$$\text{Height of transfer unit } \mathbf{H}_{OG} = \frac{G_m}{K_G'' a}$$

$$= \frac{10}{8} = 1{\cdot}25 \text{ ft}$$

$$\text{Number of transfer units } \mathbf{N}_{OG} = \frac{25{\cdot}9}{1{\cdot}25} = 20{\cdot}7 = \underline{\underline{21}} \text{ (say)}$$

## VALUES OF TRANSFER COEFFICIENTS

In calculating the size of an absorption tower, the most important single factor is the value of the transfer coefficient or the height of the transfer unit. Whilst the total flow rates of the gas and liquid streams will be fixed by the process, it is necessary to determine the most suitable flow per unit area through the column. The gas flow is limited by the fact that we must not exceed the flooding rate, and there will be a serious drop in performance if the liquid rate is very low. It is convenient to examine the effects of flow rates of the gas and liquid on the transfer coefficients, and also to investigate the influence of variables such as temperature, pressure, and diffusivity.

In the laboratory, wetted-wall columns have been used by a number of workers and they have proved valuable in determining the importance of the various factors, and have served as a basis from which somewhat similar correlations have been developed for packed towers.

### Wetted-Wall Columns

Several workers have examined the vaporisation of liquids into an air stream in a wetted-wall column, using the apparatus as outlined in Fig. 19.10. Plots of $\dfrac{d}{z_G}$ and $\dfrac{du\rho}{\mu}$ on logarithmic paper give a series of approximately straight lines showing that $\dfrac{d}{z_G} = B\, Re.^{0{\cdot}83}$

where

$d$ is the diameter of tube,

$z_G$ is the thickness of gas film,

$u$ is the gas velocity,

$\rho$ is the gas density,

$\mu$ is the gas viscosity, and

$B$ is a constant.

It is convenient to eliminate the film thickness $z_G$ in the following way.

$$k_G = \frac{D_V P}{\mathbf{R} T z_G P_{Bm}} \qquad \ldots.(19.16)$$

$$\therefore \qquad \frac{k_G \mathbf{R} T P_{Bm}}{D_V P} = \frac{1}{z_G} = \frac{B}{d} \, . \, Re.^{0\cdot 83}$$

or

$$\frac{h_D d P_{Bm}}{D_V P} = B \, Re.^{0\cdot 83} \qquad \ldots.(19.69)$$

where $h_D = k_G \mathbf{R} T$ and is a mass transfer coefficient with the driving force expressed as a molar concentration difference.

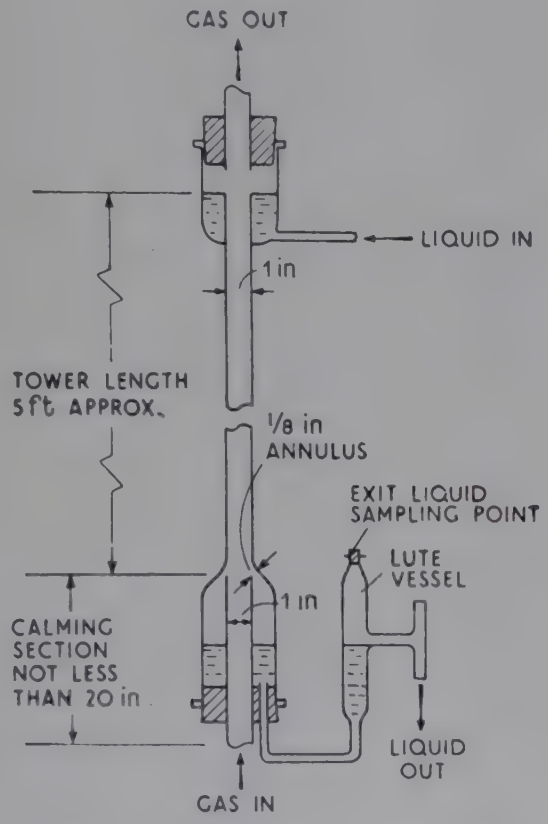

GAS OUT
LIQUID IN
1 in
TOWER LENGTH
5 ft APPROX.
1/8 in
ANNULUS
EXIT LIQUID
SAMPLING POINT
LUTE
VESSEL
1 in
CALMING
SECTION
NOT LESS
THAN 20 in
LIQUID
OUT
GAS IN

Fig. 19.10. Diagram of typical 1 in. wetted-wall column

GILLILAND and SHERWOOD's results[(12)], expressed by equation 19.69, are shown in Fig. 19.11 for a number of systems. To allow for the variation in the physical properties, the Schmidt Group *Sc.*, is introduced, and the general equation for mass transfer in a wetted-wall column is then given by

$$\frac{h_D d}{D_V} \, . \, \frac{P_{Bm}}{P} = B' \, Re.^{0\cdot 83} \, . \left(\frac{\mu}{\rho D_V}\right)^{0\cdot 44} \qquad \ldots.(19.70)$$

Values of $B'$ from 0·021 to 0·027 have been reported and a mean value of 0·023 can be taken, which then makes equation 19.70 very similar to the general heat transfer equation (6.31) for forced convection in tubes. The data shown in Fig. 19.11 are plotted as $\frac{h_D d}{D_V} \, . \, \frac{P_{Bm}}{P} \left[\frac{\mu}{\rho D_V}\right]^{-0\cdot 44}$ in Fig. 19.12, and it will be noted that they can be correlated by means of a single line.

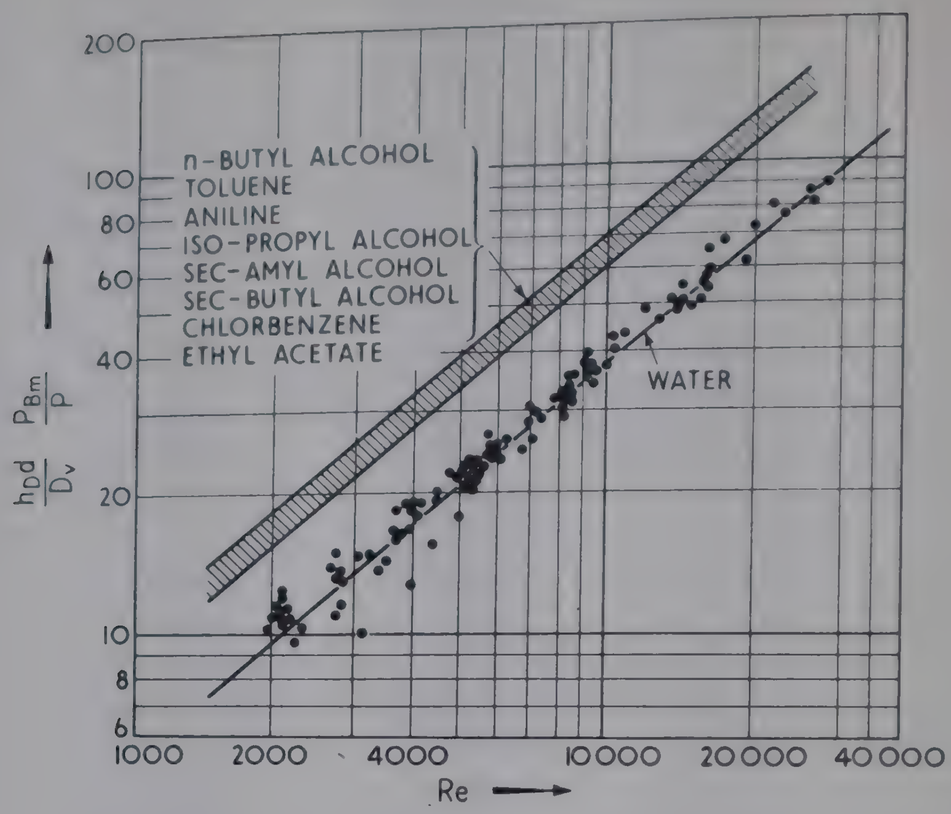


Fig. 19.11. Vaporisation of liquids in a wetted-wall column

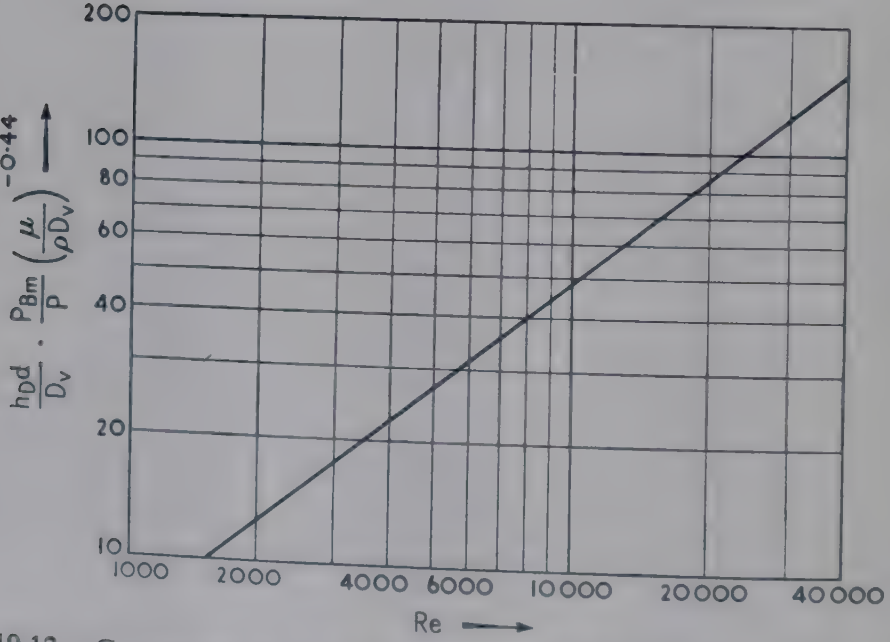


Fig. 19.12. Correlation of data on vaporisation of liquids in wetted-wall columns

The heat transfer equation is

$$\frac{hd}{k} = 0.023\, Re.^{0.8}\, Pr^{0.4} \qquad \ldots.(6.31)$$

where $h$ is the heat transfer coefficient, and

$k$ is the thermal conductivity.

In making comparison of the results from various workers, it is important to see that the inlet arrangements for the air are similar. Modifications of the inlet give rise (as found by HOLLINGS and SILVER(11)) to various values for the index on the Reynolds Group. A good calming length is necessary before the inlet to the measuring section, if results are to be reproducible.

Equation 19.70 is frequently rearranged as

$$\frac{h_D d}{D_V} \cdot \frac{P_{Bm}}{P} \cdot \frac{\mu}{du\rho}\left[\frac{\mu}{\rho D_V}\right]^{0\cdot 56} = B' Re.^{-0\cdot 17} \cdot \frac{\mu}{\rho D_V}$$

i.e.

$$\frac{h_D}{u} \cdot \frac{P_{Bm}}{P} \cdot \left[\frac{\mu}{\rho D_V}\right]^{0\cdot 56} = B' Re.^{-0\cdot 17} = j_d \qquad \ldots .(19.71)$$

(see equation 8.35 in Volume 1)

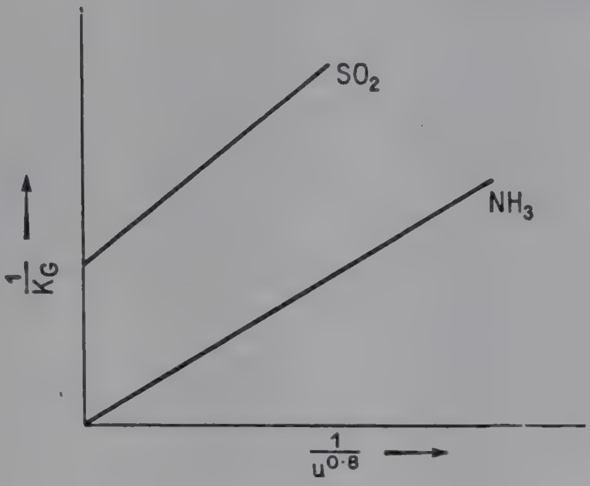


Fig. 19.13. $\frac{1}{K_G}$ versus $\frac{1}{u^{0\cdot 8}}$ for ammonia and sulphur dioxide

where $j_d$ is the $j$-factor for mass transfer as introduced by CHILTON and COLBURN(13) and discussed in Chapter 8 of Volume I. The main interest of this type of work is that it suggests that $h_D \propto G'^{0\cdot 8}$, $D_V{}^{0\cdot 56}$ and $\frac{P}{P_{Bm}}$ ; this kind of relation will be the basis for correlating work on packed towers.

***Compounding of Film Coefficients.*** Assuming $k_G \propto G'^{0\cdot 8}$, HASLAM RYAN and WEBER(5) have rearranged equation 19.24 as:

$$\frac{1}{K_G} = \frac{1}{k_G} + \frac{\mathscr{H}}{k_L} = \frac{1}{\psi u^{0\cdot 8}} + \frac{\mathscr{H}}{k_L} \qquad \ldots .(19.72)$$

If $k_L$ is assumed to be independent of the gas velocity, then a plot of $1/K_G$ versus $1/u^{0\cdot 8}$ will give a straight line with a positive intercept on the vertical axis representing the liquid film resistance $\frac{\mathscr{H}}{k_L}$. Data for ammonia and for sulphur dioxide are shown in this way in Fig. 19.13. It will be seen that in each case a straight line is obtained. The lines for ammonia pass almost through the origin showing that the liquid film resistance is very small, but the line for sulphur dioxide gives a large intercept on the vertical axis, indicating a high value of the liquid film resistance.

For a constant value of *Re.*, the film thickness $z_G$ should be independent of temperature, since $\mu/\rho D_V$ is almost independent of temperature. $k_G$ will then vary as $\sqrt{T}$. , because $D_V \propto T^{3/2}$ and $k_G \propto D_V/T$. This is somewhat difficult to test accurately since the diffusivity in the liquid phase also depends on temperature. Thus, the data of HASLAM for sulphur dioxide qualitatively support the theory for different temperatures, but the increase in value of $k_L$ masks the influence of temperature on $k_G$.

## Spray Towers

It is difficult to compare the performance of various spray towers since the type of spray distributor used will influence the results. Data from HIXSON and SCOTT(15) and others show that $K_G a$ varies as $G'^{0\cdot8}$, and is also affected by the liquor rate. Again their results for sulphur dioxide and ammonia are in fair agreement and show $K_G a$ varying as $\sqrt{D_V}$. One might expect to get more reliable data with spray columns if the liquor were introduced in the form of individual drops through a single jet into a tube full of gas. Unfortunately the drops tend to alter in size and shape and it is not possible to get the true interfacial area very accurately. This has been tried by WHITMAN(7), who found that $k_G$ for the absorption of ammonia in water was about 2·64 lb-mols/hr-ft$^2$(atmos), compared with 0·019 for the absorption of carbon dioxide in water.

Some values for the height of a transfer unit $\mathbf{H}_L$ for the stripping of oxygen from water are shown in Fig. 19.14(29). There is reason to believe that, for short heights, the spray chamber approximates closely in efficiency to that of a packed tower, but for heights greater than 4 feet the efficiency of the spray tower drops off rather rapidly. Whilst it might be thought that it would be possible to obtain a very large active interface by producing small drops, in practice it is found impossible to prevent these coalescing, and hence the effective interfacial surface falls off with height, and spray towers are not extensively used.

***Centrifugal Spray Tower.*** Fig. 19.15, taken from the work of KLEINSCHMIDT and ANTHONY(20), illustrates a new type of spray tower in which the gas stream enters tangentially, so that the liquid drops receive the centrifugal force before they are taken out of the gas stream at the top.

## Packed Towers

The majority of published results for transfer coefficients in packed towers are for rather small laboratory units of 2 to 10 in. diameter, and there is still some uncertainty in extending the data for use in industrial units. One of the great difficulties in correlating the performance of packed towers is the problem of assessing the effective wetted area for interphase transfer. It is convenient to consider separately conditions where the gas film controls the process and then where the liquid film controls. The general method of expressing results is based on that used for wetted-wall columns.

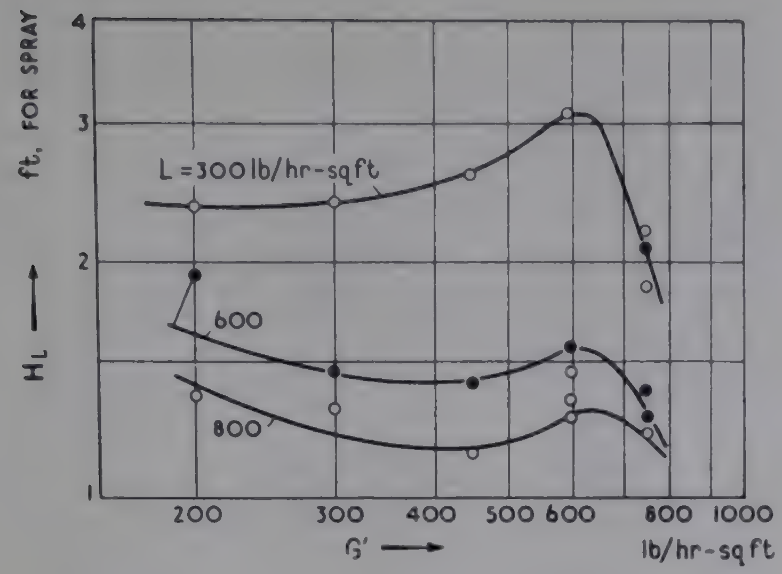


Fig. 19.14. Height of the transfer unit $H_L$ for stripping of oxygen from water

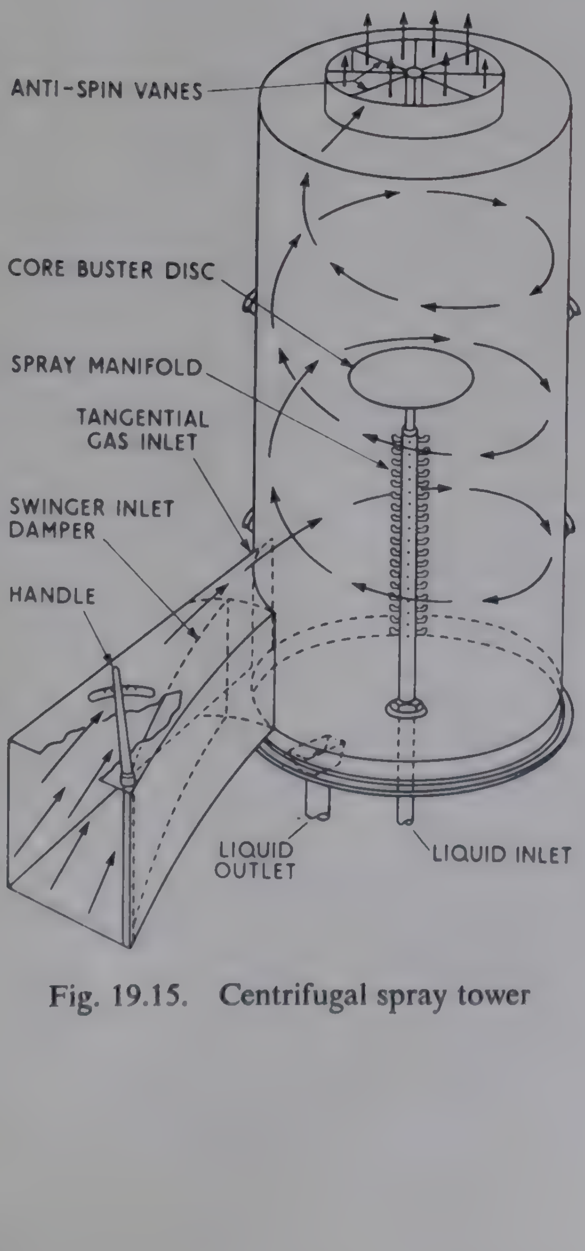

ANTI-SPIN VANES
CORE BUSTER DISC
SPRAY MANIFOLD
TANGENTIAL GAS INLET
SWINGER INLET DAMPER
HANDLE
LIQUID OUTLET
LIQUID INLET

Fig. 19.15. Centrifugal spray tower

*Gas-Film Controlled Processes.* The absorption of ammonia in water has been extensively studied by a number of workers. KOWALKE *et al.*(6) used a tower of 16 in. internal diameter with a packing 4 feet deep, and expressed their results as

$$K_G a = \alpha G'^{0\cdot8} \quad \ldots.(19.73)$$

where $K_G$ is expressed in lb-mols/hr-sq ft (unit partial pressure difference), $G'$ is in lb/hr-sq ft, and varies with the nature of the packing and the liquid rate.

TABLE 19.3. *Values of α for use in equation* 19.73

| | | | | | |
|---|---|---|---|---|---|
| Coke | 0·35–0·43 in. | 0·083 | Balls | 0·5 in. | 0·103 |
| Crushed stone | 0·25 in. | 0·116 | | 0·75 in. | 0·058 |
| | 0·5 in. | 0·105 | | 1 in. | 0·057 |
| | 0·75 in. | 0·071 | | | |

They noted that $K_G a$ increased with $L'$ for values up to 820 lb/hr-ft², after which further increase gave no significant increase in $K_G a$. It was thought that the initial increase in the coefficient was occasioned by a more effective wetting of the packing. On increasing the liquid rate so that the column approached flooding conditions, it was found that $K_G a$ fell again.

BORDEN and SQUIRES(36) using a 10 in. tower gave their results in the form

$$K_G a = 0\cdot046 G'^{0\cdot5} L'^{0\cdot4} \quad \ldots.(19.74)$$

where $G'$ varied from 55–530 lb/hr-ft², and $L'$ from 440–2050 lb/hr-ft².

FELLINGER(37) used an 18 in. diameter stoneware column in which a perforated packing support was fitted with 20 downcomers extending to within 1 in. of the bottom of the tower, and 120 risers were fitted extending 1¼ in. above the upper surface. In this way he avoided the problem of determining any entrance or exit effects. Some of his results for $\mathbf{H}_{OG}$ are shown in Table 19.4, taken from the graphs given by PERRY(1).

TABLE 19.4. *Height of the transfer unit,* $\mathbf{H}_{OG}$ *in feet*

| Raschig Rings size (in.) | $G'$ lb/hr-ft² | $\mathbf{H}_{OG}$ ($L' = 500$ lb/hr-ft²) | $\mathbf{H}_{OG}$ ($L' = 1500$ lb/hr-ft²) |
|---|---|---|---|
| ¾ | 200 | 1·2 | 0·75 |
| | 600 | 2 | 1·05 |
| 1 | 200 | 1·3 | 0·71 |
| | 600 | 2·1 | 1·1 |
| 2 | 200 | 2·0 | 1·1 |
| | 600 | 3·4 | 1·9 |

NORMAN(27) carried out experiments on the absorption of ammonia in water and on the evaporation of water in an air stream, using a tower packed with carbon slates 6 in. long × 1 in. deep × ⅛ in. thick with shallow V-notches ¼ in. deep cut in the lower edge to collect the liquid and distribute it on to the top edge of the lower grid. Such a unit gave good water distribution for values of $L'$ down to 1000 lb/hr-ft² or 2·5 lb/hr per inch perimeter of grids.

He was able to show that

$$K_G a = \alpha \,.\, G'^{0\cdot8} \qquad \ldots.(19.75)$$

where $\alpha$ depended on the liquid rate and had a value 0·0036 for $L' = 2910$ lb/hr-ft², for absorption of ammonia. For the evaporation of water, he gave the following values for equation 19.75, with $G'$ expressed in lb/hr-sq. ft.

| $L'$ lb/hr-sq. ft |  | $\alpha$ |
|---|---|---|
|  | 930 | 0 0031 |
|  | 1400 | ·0037 |
|  | 2100–2810 | ·0041 |

MOLSTAD *et al.*(23) have also measured the absorption of ammonia in water using a tower of 15⅛ in. side packed with wood grids, or with rings or saddles. Their method of treatment was to measure $K_G a$ by direct experiment, and then to calculate the value of $k_G a$ from the relation

$$\frac{1}{K_G a} = \frac{1}{k_G a} + \frac{\mathscr{H}}{k_L a} \qquad \ldots.(19.76)$$

To find the value of $k_L a$ for ammonia, they used the data obtained for the absorption of oxygen in water, where the resistance is entirely in the liquor film, and obtained the value of $k_L a$ for ammonia from the relation

$$(k_L a)_{\mathrm{NH_3}} = (k_L a)_{\mathrm{O_2}} \left(\frac{(D_L)_{\mathrm{NH_3}}}{(D_L)_{\mathrm{O_2}}}\right)^{0\cdot5} \qquad \ldots.(19.77)$$

($k_L a$ is shown to be proportional to $D_L{}^{0\cdot5}$ below.)
From these values of $(k_L a)_{\mathrm{NH_3}}$ they could then find the value of $(k_G a)_{\mathrm{NH_3}}$ using equation 19.76, and they were thus able to show that for absorption of ammonia in water some 20% of the resistance was in the liquid phase.

Both MOLSTAD(23) and VAN KREVELEN(26) have shown that the value of $\mathbf{H}_G$ varies roughly as the cube root of the packed height, presumably due to increasing maldistribution of the water with tall columns.

***Processes Controlled by Liquid-Film.*** The absorption of carbon dioxide, oxygen and hydrogen in water gives three instances in which most, if not all, of the resistance to transfer lies in the liquid phase. SHERWOOD and HOLLOWAY(19) have measured values of $k_L a$ for these systems using a tower of 20 in. diameter packed with 1½ in. rings. They were able to express their results in the form

$$\frac{k_L a}{D_L} = \beta \left[\frac{L'}{\mu_L}\right]^{0\cdot75} \left[\frac{\mu_L}{\rho_L D_L}\right]^{0\cdot50} \qquad \ldots.(19.78)$$

It should be noted that this equation has no term for characteristic length on the left hand side and therefore is not a true dimensionless equation. If values of $k_L a$ are plotted against value $L'$ on a logarithmic scale as shown in Fig. 19.16, a slope of about 0·75 is obtained for values of $L'$ from 400 to 10,000 lb./hr.-ft.². Beyond this value of $L'$, they found that $k_L a$ tended to fall because the loading point for the column was reached. These values of $k_L a$ were found to be affected by the gas rate. More recently Cooper *et al.*$^{(21)}$ established that, at the high liquor rates and low gas rates as used in practice, and with a tower packed to a depth of 86 in., the transfer rates were much lower than given by equation 19.78. This was believed to arise as a result of maldistribution at gas velocities as low as 0·1 ft/sec; in fact it is doubtful if true countercurrent conditions operate under these conditions. The results of Cooper and Sherwood are compared in Fig. 19.17, where the height of the transfer unit $\mathbf{H}_{OL}$ is plotted against the liquid rate for various gas velocities.

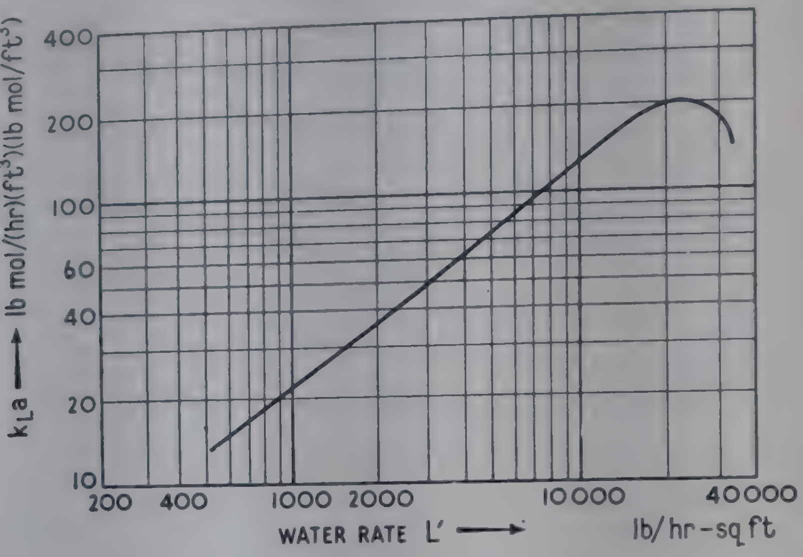


Fig. 19.16. Variation of liquid-film coefficient with liquid flow for absorption of oxygen in water

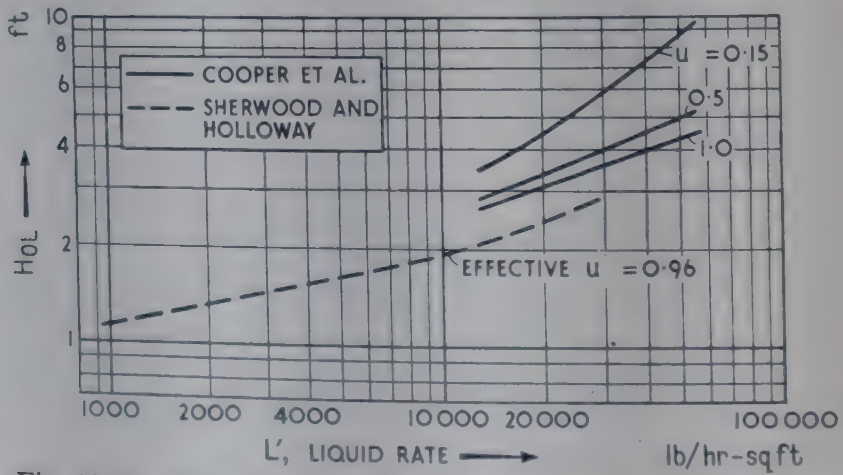


Fig. 19.17. Effect of liquid rate on height of transfer unit $\mathbf{H}_{OL}$ Comparison of results of Sherwood and Holliday, and Cooper *et al.*

## Plate Towers

Bubble cap columns, of similar construction to those described in the previous chapter for distillation, are sometimes used for gas absorption, particularly when the load is more than can be handled in a packed tower of about 3½ ft. diameter and when there is any likelihood of deposition of solids which would quickly choke a packing. Plate towers are particularly useful when the liquid rate is sufficient to flood a packed tower. The essential arrangement of such a unit is indicated in Fig. 19.18, where:

$L_m$ is the molar rate of flow per unit area of solute-free liquid,

$G_m$ is the molar rate of flow per unit area of inert gas,

$n$ refers to the plate numbered from the top downwards (and suffix $n$ refers to material leaving plate $n$),

$x$ and $y$ are the mol fractions of the absorbed component in the liquid and gas, and

$s$ is the total number of plates in the column.

It will be assumed that dilute solutions are used so that mol fractions and mol ratios are approximately equal, and that each plate is an "ideal" unit, so that the gas leaving of composition $y_n$ is in equilibrium with the liquid of composition $x_n$ leaving the plate.

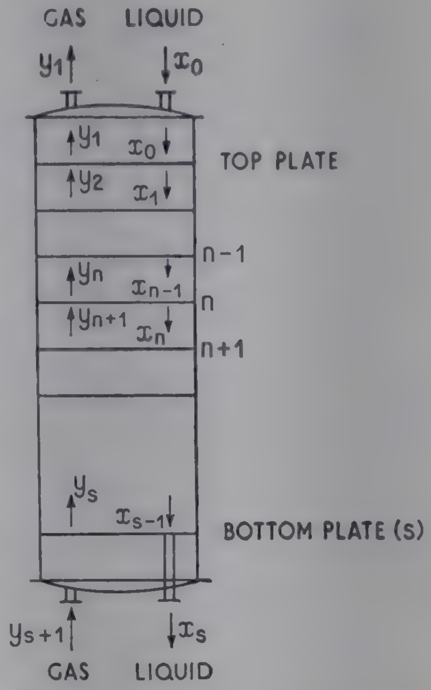


Fig. 19.18.
Plate tower—nomenclature for fluid streams

A material balance for the absorbed component from the bottom to a plane above plate $n$ will give

$$G_m y_n + L_m x_s = G_m y_{s+1} + L_m x_{n-1} \qquad \ldots.(19.79)$$

$$\therefore \qquad y_n = \frac{L_m}{G_m} x_{n-1} + y_{s+1} - \frac{L_m}{G_m} x_s \qquad \ldots.(19.80)$$

This is the equation of a straight line of slope $L_m/G_m$, relating the composition of the gas entering a plate to the liquid leaving the plate, and is known as the operating line. We can see that such a line passes through two points $A(x_s, y_{s+1})$ and $B(x_0, y_1)$ (Fig. 19.19) representing the terminal concentrations in the column. The equilibrium curve is shown in this figure as $pqr$.

Point $A$ represents conditions at bottom of the tower. The gas rising from the bottom plate is in equilibrium with a liquid of concentration $x_s$ and is shown as point 3 on the operating line. Then point 4 indicates the concentration of the liquid on the second plate from the bottom. In this way we can draw in steps to point $B$, giving the gas $y_1$ rising from top plate and liquid $x_0$ entering the top of absorber.

***Number of Plates by use of Absorption Factor.*** If the equilibrium curve can be represented by the relation $y_e = mx$, then the number of plates required for a given degree of absorption can conveniently be found by a method due to KREMSER(8) and SOUDERS and BROWN(9).

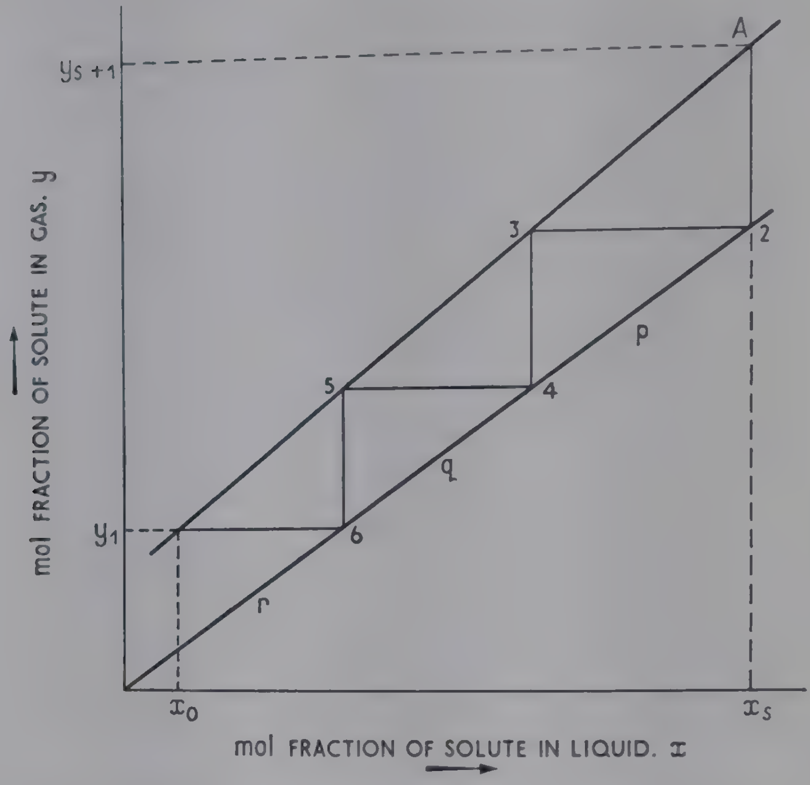
The same treatment is applicable for concentrated solutions provided concentrations are expressed as mol ratios, and if the equilibrium curve can be represented by $Y_e = mX$.

A material balance over plate $n$ gives,

$$L_m(x_n - x_{n-1}) = G_m(y_{n+1} - y_n) \qquad \ldots.(19.81)$$

But for an ideal plate, $y_n = mx_n$;

$$\therefore \qquad \frac{L_m}{mG_m}(y_n - y_{n-1}) = y_{n+1} - y_n \qquad \ldots.(19.82)$$



A
$y_{s+1}$
3
2
p
5
4
q
$y_1$
6
r
$x_0$
$x_s$
mol FRACTION OF SOLUTE IN GAS. y
mol FRACTION OF SOLUTE IN LIQUID. x

Fig. 19.19. Diagrammatic representation of changes in a plate column

This group $\dfrac{L_m}{mG_m}$, which will be taken as constant, is called the absorption factor $A$.

$$\therefore \qquad y_n = \frac{y_{n+1} + A\,.\,y_{n-1}}{1 + A} \qquad \ldots.(19.83)$$

Applying this relation to the top plate and letting $y_0$ be the mol fraction of absorbed component in the gas in equilibrium with the entering liquid, of concentration $x_0$,

$$y_1 = \frac{y_2 + Ay_0}{1 + A}$$

And for 2nd plate from top,

$$y_2 = \frac{y_3 + Ay_1}{1 + A}$$

$$= \frac{(1 + A)y_3 + Ay_2 + A^2y_0}{(1 + A)^2}$$

$$= \frac{y_3(1 + A) + A^2y_0}{A^2 + A + 1}$$

And for 3rd plate from top,

$$y_3 = \frac{y_4(1 + A + A^2) + A^3 y_0}{A^3 + A^2 + A + 1}$$

which can be written

$$y_3 = \frac{\left[\dfrac{A^3 - 1}{A - 1}\right] y_4 + A^3 y_0}{\dfrac{A^4 - 1}{A - 1}}$$

$$= \frac{(A^3 - 1)y_4 + A^3(A - 1)y_0}{A^4 - 1}$$

Proceeding thus until we reach plate $n$,

$$y_n = \frac{(A^n - 1)y_{n+1} + A^n(A - 1)y_0}{A^{n+1} - 1}$$

Putting $x_n = \dfrac{y_n}{m}$

$$x_n = \frac{(A^n - 1)y_{n+1} + A^n(A - 1)y_0}{m(A^{n+1} - 1)}$$

A material balance down to plate $n$ gives $A(y_n - y_0) = y_{n+1} - y_1$ (see equation 19.83).

$$\therefore \quad \frac{y_{n+1} - y_1 + A \cdot y_0}{A} = y_n = \frac{(A^n - 1)y_{n+1} + A^n(A - 1)y_0}{A^{n+1} - 1}$$

$$\therefore \quad y_1 = y_{n+1}\left[\frac{A - 1}{A^{n+1} - 1}\right] + y_0\left[\frac{A^{n+1} - A}{A^{n+1} - 1}\right]$$

Since $\dfrac{A - 1}{A^{n+1} - 1} = 1 - \dfrac{A^{n+1} - A}{A^{n+1} - 1}$,

$$y_1 = y_{n+1} + (y_0 - y_{n+1}) \frac{(A^{n+1} - A)}{A^{n+1} - 1}$$

or

$$\frac{y_{n+1} - y_1}{y_{n+1} - y_0} = \frac{A^{n+1} - A}{A^{n+1} - 1}$$

But $y_{n+1} - y_1$ = actual change in composition of gas,
and $y_{n+1} - y_0$ = maximum possible change in composition of gas, i.e., if gas leaving absorber is in equilibrium with entering liquid.

If $y_1$, $y_{s+1}$, $x_s$, $x_0$ are the terminal concentrations, then $y_0 = mx_0$ and we get

$$\frac{y_{s+1} - y_1}{y_{s+1} - mx_0} = \frac{\left(\dfrac{L_m}{mG_m}\right)^{s+1} - \dfrac{L_m}{mG_m}}{\left(\dfrac{L_m}{mG_m}\right)^{s+1} - 1} \qquad \ldots.(19.84)$$

This equation is conveniently represented as suggested by SOUDERS and BROWN by Fig. 19.20, and it is easy to use such a diagram to determine the number of plates required.

We can now see that a high degree of absorption can be obtained, either by using a large number of plates, or by using a high absorption factor $\frac{L_m}{mG_m}$. Since $m$ is fixed by the system, this means that $L_m/G_m$ must be large if a high degree of absorption is to be obtained, but this will result in a low value of $x$ for the liquid leaving at the bottom. This problem is to some extent met by recirculating the liquor over the tower, but the advantages of a countercurrent flow system are then lost. COLBURN has suggested (see earlier) that a value of $\frac{mG_m}{L_m}$ of about 0·7–0·8 is probably the most economical. (See page 720). i.e. $\frac{L_m}{mG_m} = 1{\cdot}3$.

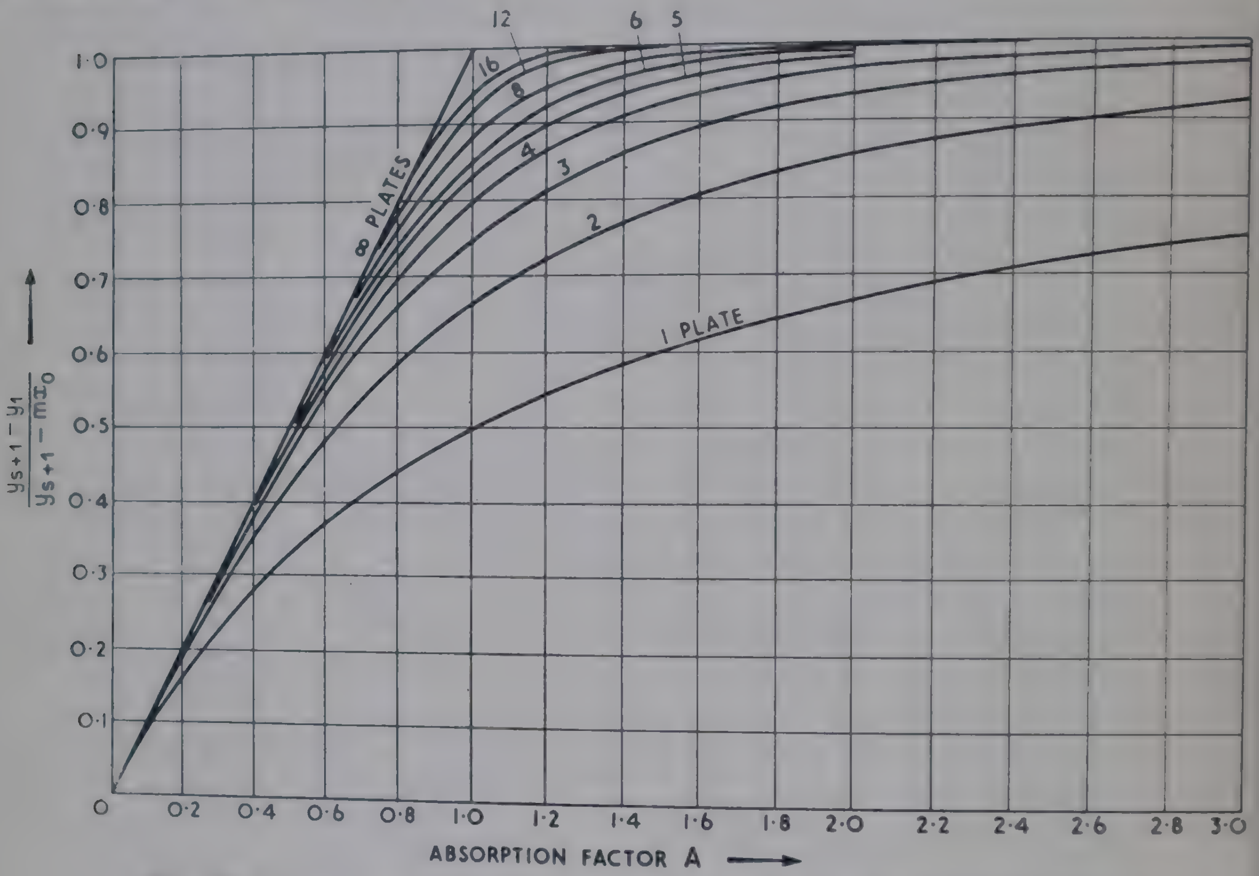


Fig. 19.20. Graphical representation of the effect of the absorption factor and the number of plates on the degree of absorption

It is important to note that, if $\frac{L_m}{mG_m}$ is less than 1, then a very large number of plates are required to achieve a high recovery, and even an infinite number will not give complete recovery. $\frac{L_m}{mG_m}$ is the ratio of the slope of the operating line $\frac{L_m}{G_m}$ to the slope of the equilibrium curve $m$, so that if $\frac{L_m}{G_m} < m$, i.e. $\frac{L_m}{mG_m} < 1$, then the operating line will never cut the equilibrium curve and the gas leaving the top of the column will not therefore reach equilibrium with the entering liquid.

## MISCELLANEOUS EQUIPMENT

### The Use of Vessels with Agitators

A gas may be dissolved in a liquid by dispersing it through holes in a pipe immersed in the liquid which is stirred with some form of agitator (Fig. 19.21). This type of equipment will give only one theoretical stage per unit, but it often provides a useful method of saturating a liquid with a gas. COOPER *et al.*(25) have studied the absorption of oxygen from the air in an aqueous solution of sodium sulphite. They used simple vessels of 6 to 96 in. diameter fitted with four simple baffles, and introduced the air just below the agitator which was a vaned disc or flat-paddle. They found that the absorption coefficient $K_G a$ varied almost directly with $p_V$ the power input per unit volume. For constant values of $p_V$, they obtained the relation

$$K_G a = \gamma u_s^{0\cdot67} \qquad \ldots.(19.85)$$

where $u_s$ is the superficial gas velocity based on the volume of gas at inlet and the cross-section of tank. A general correlation was obtained by plotting $\dfrac{K_G a}{u_s^{0\cdot67}}$ against the power input per unit volume, as shown in Fig. 19.22 taken from their work. AYERST(33) has given some data on the use of this type of unit for the absorption of carbon dioxide from ammoniacal solutions.

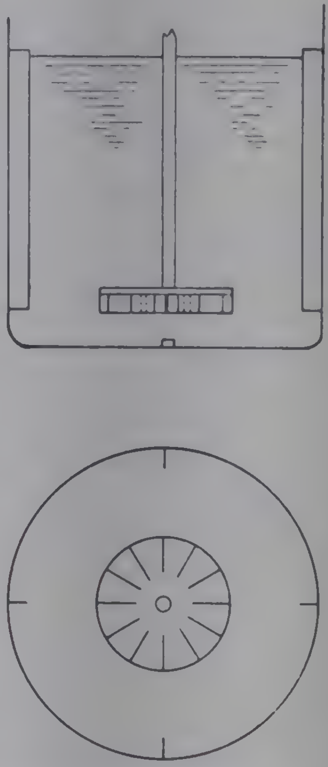


Fig. 19.21. Vessel with vaned-disc agitator

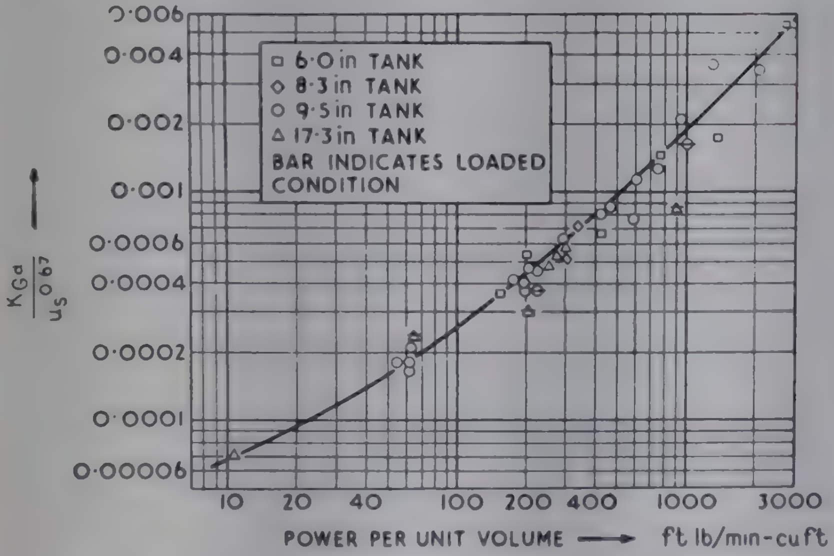


Fig. 19.22. General correlation of data for vessel (height = diameter) with vaned-disc agitator

### The Centrifugal Absorber

In an attempt to obtain the benefits of repeated spray formations, a centrifugal type absorber has been developed from the ideas of PIAZZA for a still-head. The principle of the unit is indicated in Fig. 19.23(34). A set of stationary concentric rings intermesh with a second set of rings attached to a rotating plate. Liquid fed to the centre of the plate is carried up the first ring, splashes over to the baffle and falls into the trough between the rings. It then runs up the second ring and in a similar way passes from ring to ring through the unit. The gas stream can be introduced at the top to give co-current flow, or at the bottom if countercurrent flow is desired. Some of the features of this unit are discussed by AHMED(38), who found that the depth of the ring was not very important and that most of the transfer took place as the gas mixed with the liquid spray leaving the top of the rings. CHAMBERS and WALL(34) have given some particulars of the performance of the 20-in. diameter unit shown in Fig. 19.24, for the absorption of carbon dioxide from air containing 10–15% of carbon

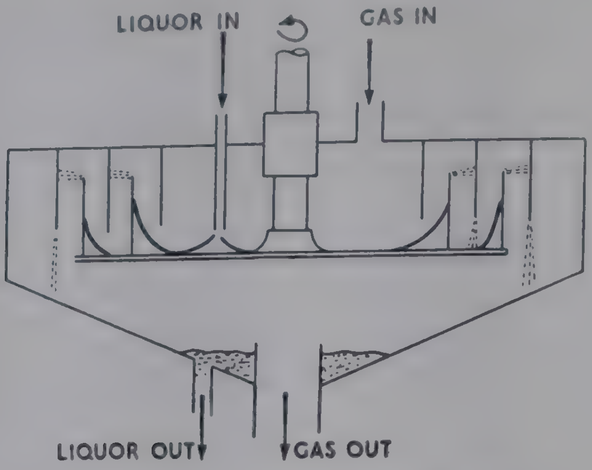


Fig. 19.23. The centrifugal absorber

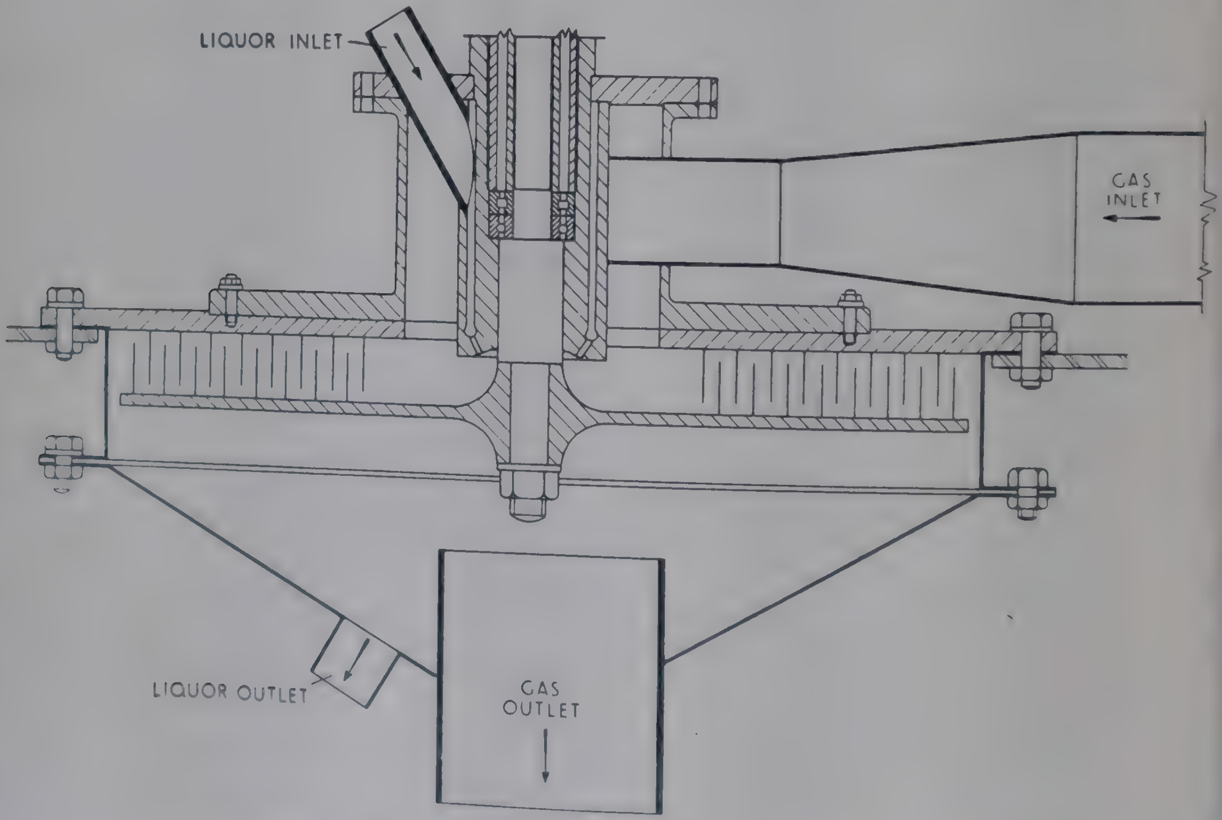


Fig. 19.24. Details of 20-in. diameter centrifugal absorber

dioxide, using mono-ethanolamine solution. Some figures are given in Table 19.5, below.

TABLE 19.5. *Preliminary results for absorption in large unit*

| Gas flow (cu. ft/hr) | Liquor flow (gal/hr) | $CO_2$ in gas % | | Absorption (lb/hr) |
|---|---|---|---|---|
| | | In | Out | |
| 2,000 | 85 | 16·3 | 2·3 | 35·3 |
| 3,000 | 85 | 15·8 | 4·5 | 43·7 |
| 4,000 | 85 | 14·3 | 6·6 | 40·5 |
| 5,000 | 85 | 16·3 | 8·7 | 51·2 |
| 4,100 | 75 | 14·8 | 2·2 | 65·3 (new monoethanolamine) |

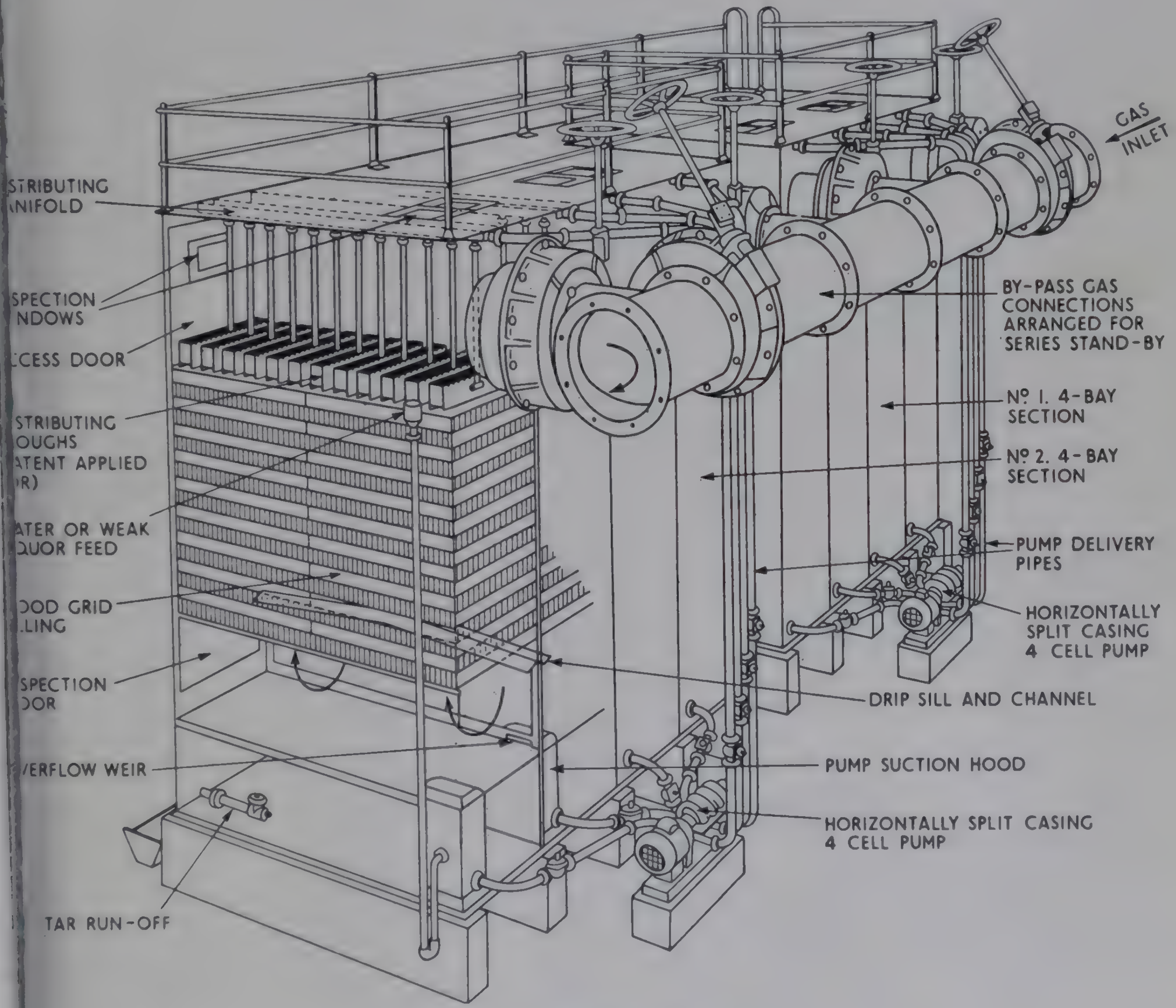

GAS INLET
STRIBUTING
NIFOLD
SPECTION
NDOWS
CESS DOOR
STRIBUTING
OUGHS
TENT APPLIED
R)
TER OR WEAK
QUOR FEED
OOD GRID
LING
SPECTION
OOR
ERFLOW WEIR
TAR RUN-OFF
BY-PASS GAS CONNECTIONS ARRANGED FOR SERIES STAND-BY
Nº 1. 4-BAY SECTION
Nº 2. 4-BAY SECTION
PUMP DELIVERY PIPES
HORIZONTALLY SPLIT CASING 4 CELL PUMP
DRIP SILL AND CHANNEL
PUMP SUCTION HOOD
HORIZONTALLY SPLIT CASING 4 CELL PUMP

Fig. 19.25. Whessoe gas scrubber

### The Whessoe Gas Scrubber

In those cases where the ratio of liquid to gas is low, the multistage washer developed by Whessoe has proved useful, particularly in the coal gas industry for removal of ammonia and naphthalene. In such a unit (Fig. 19.25), the solvent is recirculated at a high rate in each section, and the gas makes a single passage. The units are conveniently arranged in groups of four with a multicell-pump for the circulation of the liquid at each stage. A typical unit handling 3 million cubic feet of gas per day has 4 stages and is 8 ft. 2 in. long, by 4 ft. 9 in. wide, by 12 ft. 11 in. high, and requires a motor of only 2½ H.P. to drive the pump.

## SOME DESIGN CONSIDERATIONS

When designing a unit for gas absorption, a rough indication of the size can be obtained by deduction from known transfer coefficients for somewhat similar systems, but it is desirable to carry out an experiment in a small laboratory unit. This unit will show up the influence of most of the variables but it is at present very difficult to scale up with certainty from a small laboratory packed column to a large commercial unit. This difficulty really arises from the fact that we cannot maintain conditions of similarity between the small and large packing. It is therefore desirable to do pilot scale experiments in packed towers using the packing which will be used in the larger unit. This is particularly desirable where a chemical reaction takes place.

## ABSORPTION ASSOCIATED WITH CHEMICAL REACTION

In the instances so far considered, the process of absorption of the gas in the liquid is really entirely a physical one. There are, however, a number of cases in which the gas, on absorption, reacts chemically with a component of the liquid phase. Thus, in the absorption of carbon dioxide by caustic soda, the carbon dioxide reacts directly with the caustic soda and the process of mass transfer is thus made much more complicated. Again, when carbon dioxide is absorbed in ethanolamine solution, there is direct chemical reaction between the amine and the gas. In such processes the conditions in the gas phase are similar to those already discussed, but in the liquid phase there is a liquid film followed by a reaction zone. The process of diffusion and chemical reaction can still be represented by an extension of the film theory by a method due to HATTA(10).

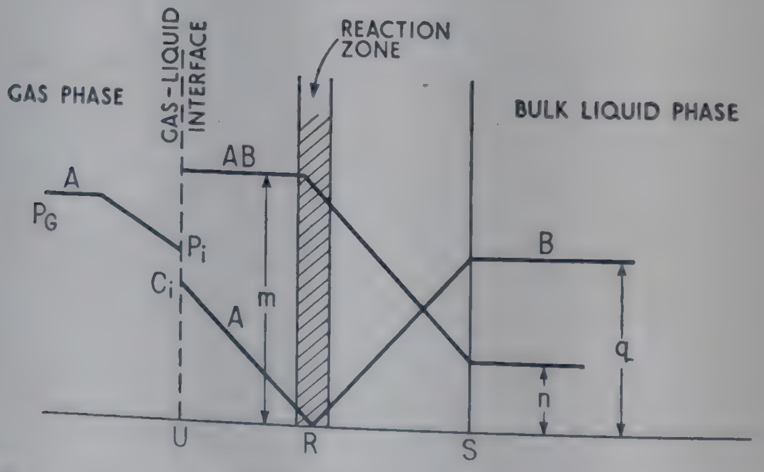


Fig. 19.26. Absorption with chemical reaction—concentration profiles

In the case considered, the chemical reaction is irreversible and of the type in which a solute gas *A* is absorbed from a mixture by a substance *B* in the liquid phase, which combines with *A* according to the equation $A + B \rightarrow AB$. As the gas approaches the liquid interface, it dissolves and reacts at once with *B*. The new product *AB*, thus formed, diffuses towards the main body of the liquid. The concentration of *B* at the interface falls; this results in diffusion of *B* from the bulk of the liquid phase to the interface. Since the chemical reaction is rapid, *B* is removed very quickly, so that it is necessary for the gas *A* to diffuse through part of the liquid film before meeting *B*. There is thus a zone of reaction between *A* and *B* which moves away from the gas-liquid interface, taking up some position towards the bulk of the liquid. The final position of this reaction zone will be such that the rate of diffusion of *A* from the gas-liquid interface is equal to the rate of diffusion *B* from the main body of the liquid. When this condition has been reached, the concentrations of *A*, *B*, and *AB* can be indicated as in Fig. 19.26, where the concentrations are shown as ordinates and the position of a plane relative to the interface is abscissae. In the diagram, the plane of the interface between gas and liquid is shown by *U*, the reaction zone by *R*, and the outer boundary of liquid film by *S*. Then *A* diffuses through the gas film as a result of the driving force $P_G - P_i$, and diffuses to the reaction zone as a result of the driving force $C_i$ in the liquid phase. The component *B* diffuses from the main body of the liquid to the reaction zone under a driving force *q*, and the non-volatile product *AB* diffuses back to the main bulk of the liquid under a driving force $m - n$.

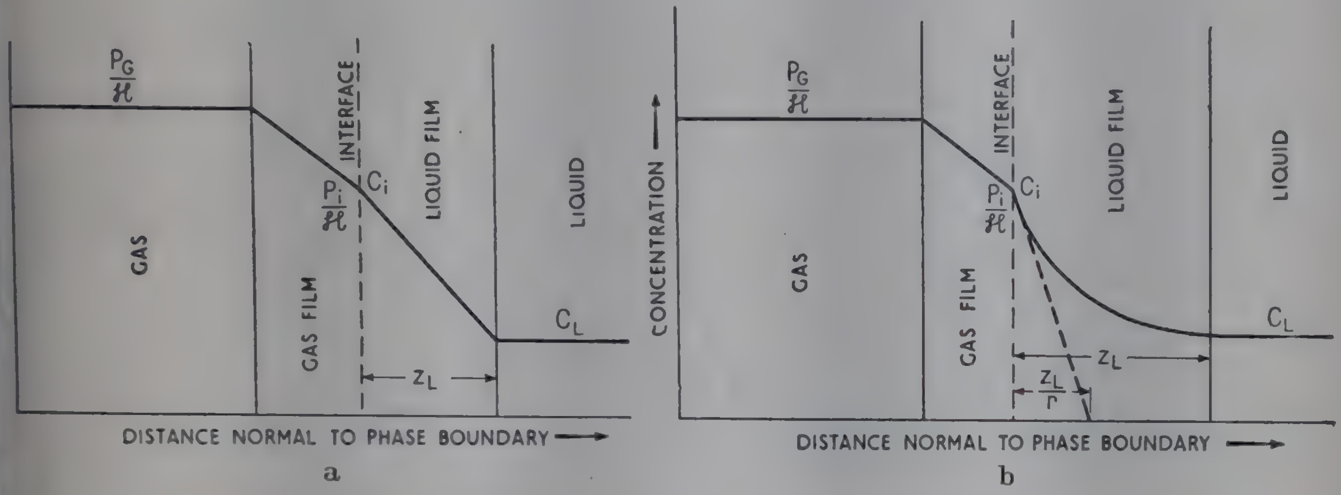

$\frac{P_G}{\mathcal{H}}$
GAS
GAS FILM
INTERFACE
$\frac{P_i}{\mathcal{H}}$
$C_i$
LIQUID FILM
LIQUID
$C_L$
$z_L$
DISTANCE NORMAL TO PHASE BOUNDARY
a
CONCENTRATION
$\frac{P_G}{\mathcal{H}}$
GAS
GAS FILM
INTERFACE
$\frac{P_i}{\mathcal{H}}$
$C_i$
LIQUID FILM
LIQUID
$C_L$
$z_L$
$\frac{z_L}{r}$
DISTANCE NORMAL TO PHASE BOUNDARY
b

Fig. 19.27. Concentration profiles for absorption
(a) without chemical reaction
(b) with chemical reaction

The difference between a physical absorption, and one in which a chemical reaction occurs, can also be shown by Figs. 19.27*a* and 19.27*b*, taken from a paper by VAN KREVELEN(32). Diagram (*a*) shows the normal concentration profile for physical absorption whilst (*b*) shows the profile modified by the chemical reaction.

For transfer in the gas phase,

$$N_A' = k_G(P_G - P_i) \quad \ldots.(19.86)$$

and in the liquid phase,

$$N_A' = k_L(C_i - C_L) \quad \ldots.(19.87)$$

The effect of the chemical reaction is to accelerate the removal of $A$ from the interface, and we can suppose that it is now $r$ times as great so that

$$N_A'' = rk_L(C_i - C_L) \quad \ldots.(19.88)$$

In Fig. 19.27(*a*), the concentration profile through the liquid film of thickness $z_L$ is represented by a straight line such that $k_L = \frac{D_L}{z_L}$. In (*b*), component $A$ is removed by chemical reaction, so that the concentration profile is curved. The dotted line gives the concentration profile if—for the same rate of absorption—$A$ was removed only by diffusion. The effective diffusion path is thus $\frac{1}{r}$ times the total film thickness $z_L$.

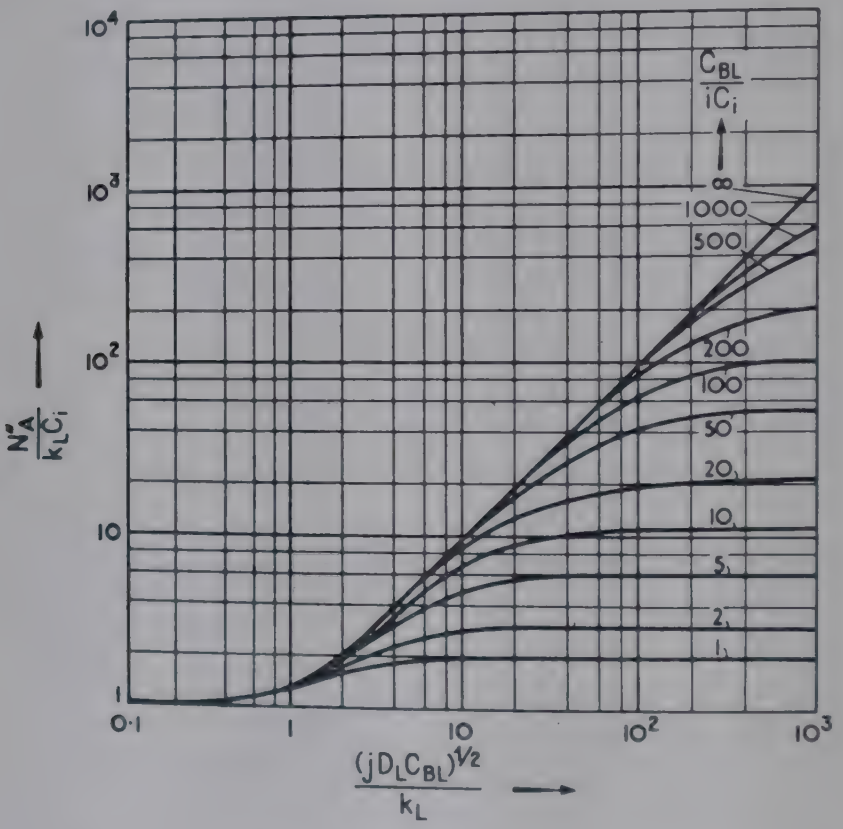


Fig. 19.28. $\frac{N_A''}{k_L C_i}$ versus $\frac{(jD_L C_{BL})^{\frac{1}{2}}}{k_L}$ for various values of $\frac{C_{BL}}{iC_i}$

$$\therefore \quad N_A'' = \frac{rD_L}{z_L}(C_i - C_L) = rk_L(C_i - C_L) \quad \ldots.(19.89)$$

Van Krevelen has shown that this factor $r$ can be related to $C_i$, $D_L$, $k_L$, to the concentration of $B$ in the bulk liquid $C_{BL}$, and to the reaction rate constant $j$. His suggested relationship is shown in Fig. 19.28, in which $r$, i.e. $\frac{N_A''}{k_L C_i}$, is plotted versus $\frac{(jD_L C_{BL})^{\frac{1}{2}}}{k_L}$, for various values of $\frac{C_{BL}}{iC_i}$ (where $i$ is the number of mols of $B$ combining with 1 mol of $A$). This diagram brings out three conditions:

(1) If $j$ is very small, $r \simeq 1$, and we have conditions of physical absorption.

(2) If $j$ is very large, $r \simeq \frac{C_{BL}}{iC_i}$, and the rate of the process is determined by the transport of $B$ towards the phase boundary.

(3) At moderate values of $j$, $r \simeq \frac{(jD_L C_{BL})^{\frac{1}{2}}}{k_L}$, and the rate of the process is determined by the rate of the chemical reaction.

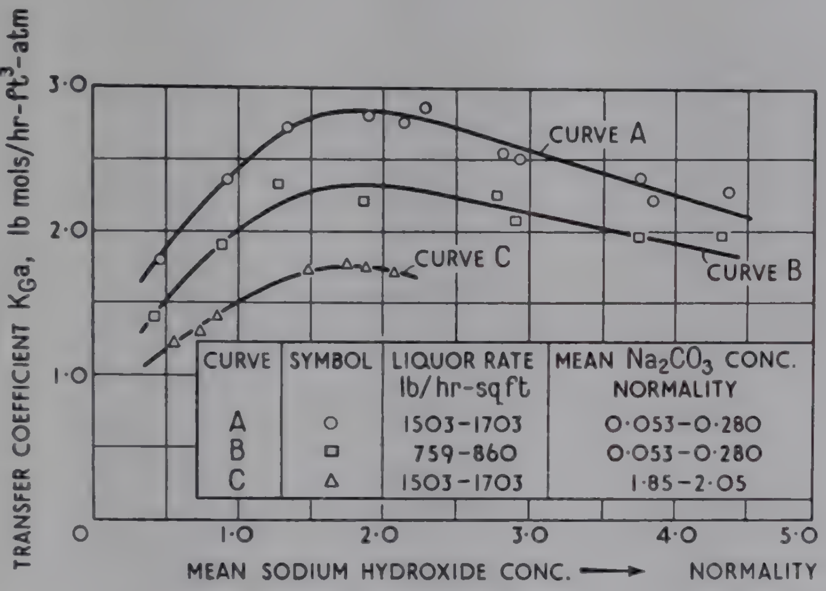


Fig. 19.29. Absorption of carbon dioxide in sodium hydroxide solution $G' = 188\text{-}193$ lb/hr-sq ft. Temperature = 25°C

As an illustration of combined absorption and chemical reaction, we can take the results of Tepe and Dodge[24] on the absorption of carbon dioxide by sodium hydroxide solution. They used a 6 in. diameter tower filled to a 36 in. depth with ½ in. carbon Raschig rings. Some of their results are indicated in Fig. 19.29. $K_Ga$ increases rapidly with increasing sodium hydroxide concentration up to a value of about 2 normal. Changes in the gas rate were found to have negligible effect on $K_Ga$, indicating that the major resistance to absorption was in the liquid phase. The influence of the liquid rate was rather low, and corresponded to $L'^{0 \cdot 28}$. It may be assumed that, in this case, the final rate of the process is controlled by the resistance to diffusion in the liquid, by the rate of the chemical reaction, or by both together.

CRYDER and MALONEY(22) have presented data on the absorption of carbon dioxide in diethanolamine solution, using an 8 in. tower filled with 3/4 in. rings. Some of their results are indicated in Fig. 19.30. The coefficient $K_G a$ is found to be independent of the gas rate but to increase with the liquid rate, as expected in a process controlled by the resistance in the liquid phase.

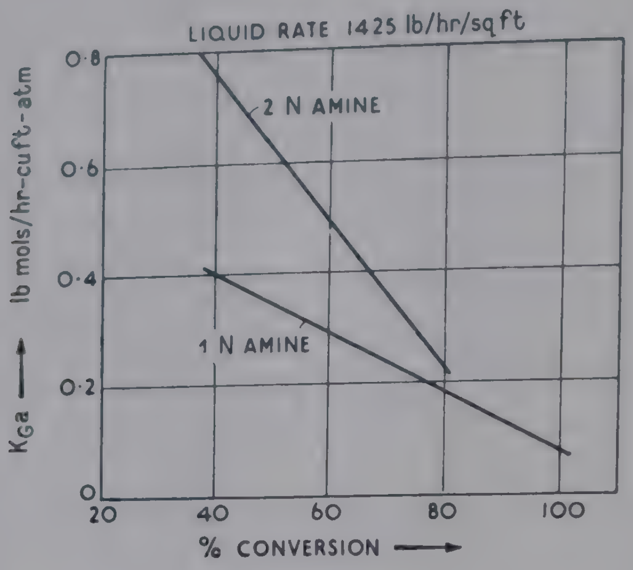


Fig. 19.30. Absorption of carbon dioxide in diethanolamine solutions

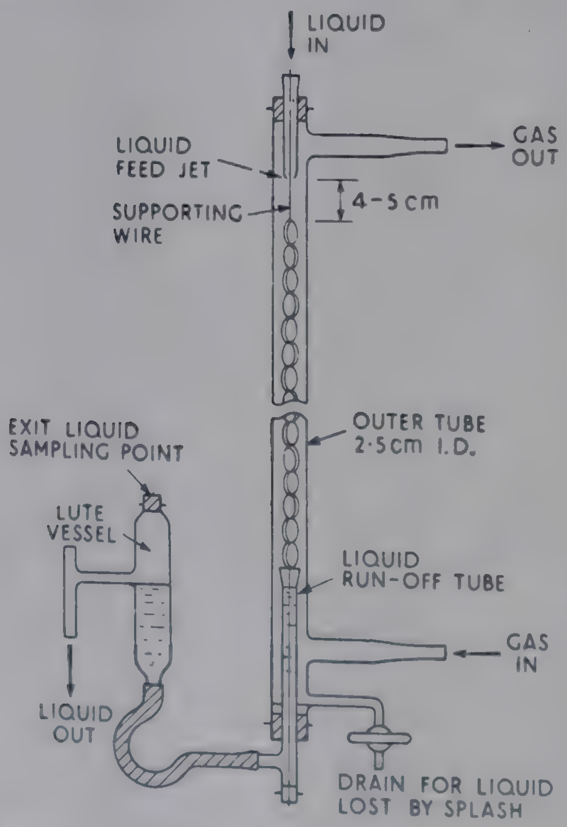

LIQUID IN
GAS OUT
LIQUID FEED JET
SUPPORTING WIRE
4–5 cm
EXIT LIQUID SAMPLING POINT
OUTER TUBE 2·5 cm I.D.
LUTE VESSEL
LIQUID RUN-OFF TUBE
GAS IN
LIQUID OUT
DRAIN FOR LIQUID LOST BY SPLASH

Fig. 19.31. Small disc tower for absorption tests

It is very difficult at present to deduce the size of tower required for an absorption combined with a chemical reaction, and an experiment in some pilot plant should be carried out in all cases of this kind. STEPHENS and MORRIS(30) have recently suggested a small disc type tower illustrated in Fig. 19.31, which they have successfully used for preliminary experiments of this kind. They found that a simple wetted-wall column was unsatisfactory where chemical reactions took place. In their unit a series of discs, supported by means of a wire, is arranged one on top of the other as shown.

## ABSORPTION ACCOMPANIED BY THE LIBERATION OF HEAT

In some absorption processes, especially where a chemical reaction occurs, there is a liberation of heat. This generally gives rise to an increase in the temperature of the liquid, with the result that the position of the equilibrium curve is adversely affected. The rise in temperature, and hence the change in the position of the equilibrium curve, can be calculated by a stagewise procedure, by considering the absorption of successive fractions of material. If the amount of heat liberated is very large, it may be necessary to cool the liquid. This is most conveniently done in a plate column, either with heat exchangers connected between consecutive plates, or with cooling coils on the plate, as shown in Fig. 19.32.

## MECHANISM OF MASS TRANSFER

The analysis of the process of absorption so far given is based on the two-film theory of WHITMAN(4). It is supposed that the two films have negligible capacity, but offer all the resistance to mass transfer. Any turbulence disappears at the interface or free surface, and the flow is thus considered to be laminar and parallel to the surface. For convenience, we assume that this film can be regarded as a layer of thickness $z_L$ and the mechanism is one of diffusion through this layer, the rate of transfer being given by

$$N_A' = k_L(C_i - C_L) \qquad \ldots.(19.20)$$

where $C_i$ is the concentration of dissolved gas at the surface, and

$C_L$ is the concentration of the dissolved gas in the bulk of the liquid.

As we have seen, we can write equation 19.20 as

$$N_A' = (C_i - C_L) \, . \, \frac{D_L}{z_L} \qquad \ldots.(19.90)$$

where $D_L$ is the diffusivity of the dissolved component in the liquid.

An alternative theory has been put forward by HIGBIE(16), and later extended by DANCKWERTS(31, 35), in which the slow moving film is not envisaged. HIGBIE supposed that the liquid surface was composed of a large number of small elements each of which is exposed to the gas phase for an equal interval of time. At the end of this time interval, these elements are replaced by fresh elements arising from the bulk of the liquid. During the period in which the elements are exposed to the gas phase, transfer takes place in the same way as in the previous theory. If an element of surface absorbs an amount $Q$ of gas per unit area during its brief exposure $t$, the average rate of absorption will be $\frac{Q}{t}$. The rate of transfer is shown to be given by

$$N_A' = (C_i - C_L)2\sqrt{\frac{D_L}{\pi t}} \qquad \ldots.(19.91)$$

DANCKWERTS has modified this theory by suggesting that each element of surface is not exposed for the same period, but that the times of exposure of the elements are random. On this basis he obtained

$$N_A' = (C_i - C_L)\sqrt{D_L f} \qquad \ldots.(19.92)$$

where $f$ is the fraction of the surface renewed per unit time.
The average absorption rate is

$$f\int_{t=0}^{t=\infty} e^{-ft}\,\mathrm{d}Q \qquad \ldots.(19.93)$$

All three of these proposals give $N_A' \propto (C_i - C_L)$ so that they do not directly enable a decision to be made between the theories. We can see, however, that on the HIGBIE-DANCKWERTS theory $N_A' \propto \sqrt{D_L}$ so that we should expect $z_L \propto \sqrt{D_L}$. DANCKWERTS(31) applied this theory to the problem of absorption coupled with chemical reaction but, although in this case the three proposals give somewhat different results, it has not been possible to distinguish between them.

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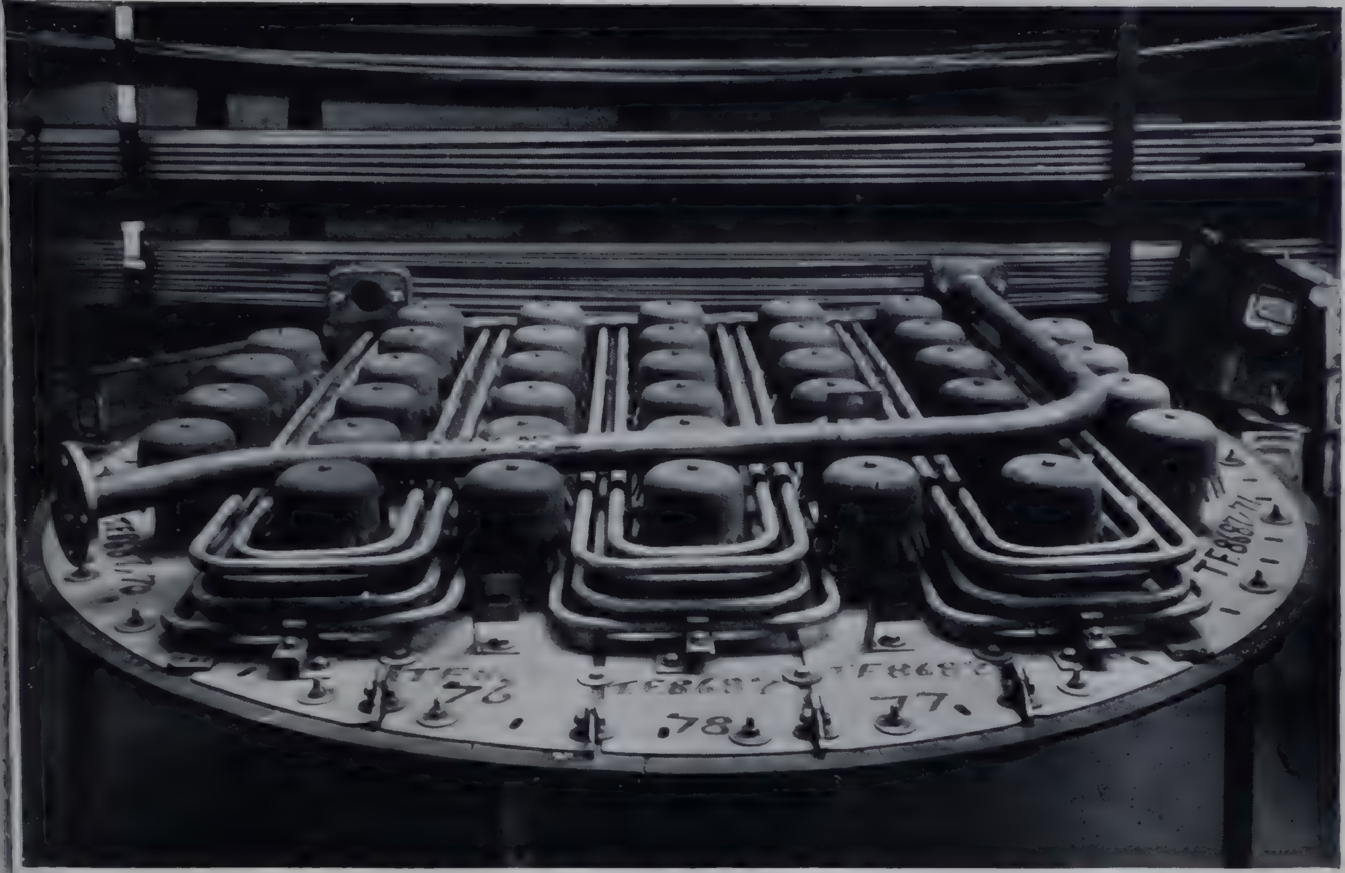

Fig. 19.32. Glitsch "truss type" bubble tray in stainless steel for 6 ft 3 in. absorption column

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## LIST OF SYMBOLS USED IN CHAPTER 19

| | | |
|---|---|---|
| $A$ | Absorption factor | — |
| $a$ | Surface of interface per unit volume of column | $L^{-1}$ |
| $B$ | A constant | — |
| $B'$ | A constant | — |
| $C$ | Molar concentration | $ML^{-3}$ |
| $C_A, C_B$ | Molar concentrations of $A$, $B$ | $ML^{-3}$ |
| $C_{BL}$ | Molar concentration of $B$ in bulk of liquid phase | $ML^{-3}$ |
| $C_e$ | Molar concentration in liquid phase in equilibrium with partial pressure $P_G$ in gas phase | $ML^{-3}$ |
| $C_i$ | Molar concentration at interface | $ML^{-3}$ |
| $C_L$ | Molar concentration in bulk of liquid | $ML^{-3}$ |
| $C_T$ | Total molar concentration | $ML^{-3}$ |
| $c$ | Constant term in equation of equilibrium line | — |
| $D_L$ | Liquid phase diffusivity | $L^2T^{-1}$ |
| $D_V$ | Vapour phase diffusivity | $L^2T^{-1}$ |
| $d$ | Column diameter | L |
| $f$ | Fraction of surface renewed per unit time | $T^{-1}$ |
| $G_m$ | Molar rate of flow of inert gas per unit cross-section | $ML^{-2}T^{-1}$ |
| $G'$ | Gas flow rate (mass) per unit cross-section | $ML^{-2}T^{-1}$ |
| **H** | Height of transfer unit | L |
| $h$ | Heat transfer coefficient | $ML^{-2}T^{-1}$ |
| $h_D$ | Mass transfer coefficient | $LT^{-1}$ |
| $\mathscr{H}$ | Henry's constant | $L^2T^{-2}$ |
| $i$ | Number of mols of $B$ reacting with 1 mol of $A$ | — |
| $j$ | Reaction rate constant | $M^{-1}L^3T^{-1}$ |
| $j_d$ | $j$-factor for mass transfer | — |
| $K_G$ | Overall gas-phase transfer coefficient | $L^{-1}T$ |
| $K_L$ | Overall liquid-phase transfer coefficient | $LT^{-1}$ |
| $K''_G$ | Overall gas-phase transfer coefficient in terms of mol fractions | $ML^{-2}T^{-1}$ |
| $K''_L$ | Overall liquid-phase transfer coefficient in terms of mol fractions | $ML^{-2}T^{-1}$ |
| $k$ | Thermal conductivity | $ML^{-1}T^{-1}$ |
| $k_G$ | Gas film transfer coefficient $\left(\dfrac{D_V . P}{\mathbf{R}Tz_G P_{Bm}}\right)$ | $L^{-1}T$ |
| $k'_G$ | Gas film transfer coefficient $\left(\dfrac{D_V}{\mathbf{R}Tz_G}\right)$ | $L^{-1}T$ |
| $k''_G$ | Gas film transfer coefficient in terms of mol fractions | $ML^{-2}T^{-1}$ |
| $k_L$ | Liquid film transfer coefficient | $LT^{-1}$ |
| $k''_L$ | Liquid film transfer coefficient in terms of mol fractions | $ML^{-2}T^{-1}$ |
| $L_m$ | Molar rate of flow of solute-free liquor per unit cross-section | $ML^{-2}T^{-1}$ |
| $L'$ | Liquid flow rate (mass) per unit cross-section | $ML^{-2}T^{-1}$ |
| $m$ | Slope of equilibrium line | — |
| $N_A, N_B$ | Molar rate of diffusion of $A$, $B$ per unit area | $ML^{-2}T^{-1}$ |
| $N'_A, N'_B$ | Molar rate of absorption of $A$, $B$ per unit area | $ML^{-2}T^{-1}$ |
| $N''_A$ | Molar rate of absorption of $A$ per unit area with chemical reaction | $ML^{-2}T^{-1}$ |
| **N** | Number of transfer units | — |
| $n$ | Number of plates from top | — |
| $P$ | Total pressure | $ML^{-1}T^{-2}$ |
| $P_A, P_B$ | Partial pressures of $A$ and $B$ | $ML^{-1}T^{-2}$ |
| $P_{Bm}$ | Logarithmic mean value of $P_B$ | $ML^{-1}T^{-2}$ |
| $P_e$ | Partial pressure of $A$ in equilibrium with concentration $C_l$ in liquid phase | $ML^{-1}T^{-2}$ |
| $P_G$ | Partial pressure of $A$ in bulk of gas phase | $ML^{-1}T^{-2}$ |
| $P_i$ | Partial pressure of $A$ at interface | $ML^{-1}T^{-2}$ |

| | | |
|---|---|---|
| $p_V$ | Power input per unit volume | $ML^{-1}T^{-3}$ |
| $Q$ | Molar absorption in time $t$ | $ML^{-2}$ |
| R | Universal gas constant | $L^2T^{-2}\theta^{-1}$ |
| $r$ | Ratio of effective film thickness for absorption without and with chemical reaction | — |
| $S$ | Cross-sectional area of column | $L^2$ |
| $s$ | Total number of plates in column | — |
| $T$ | Absolute temperature | $\theta$ |
| $t$ | Time | T |
| $u$ | Gas velocity | $LT^{-1}$ |
| $u_s$ | Superficial gas velocity based on inlet conditions | $LT^{-1}$ |
| $V$ | Volume of packed section of column | $L^3$ |
| $X$ | Mols of solute gas $A$ per mol of solvent in liquid phase | — |
| $x$ | Mol fraction of $A$ in liquid phase | — |
| $Y$ | Mols of solute gas $A$ per mol of inert gas $B$ in gas phase | — |
| $y$ | Mol fraction of $A$ in gas phase | — |
| $Z$ | Height of packed column | L |
| $z$ | Distance in direction of mass transfer | L |
| $z_G$ | Thickness of gas film | L |
| $z_L$ | Thickness of liquid film | L |
| $\alpha$ | A coefficient | $M^{-0.8}L^{-0.4}T^{1.8}$ |
| $\beta$ | A coefficient | $L^{-5/4}$ |
| $\gamma$ | A coefficient | $L^{-8/3}T^{5/3}$ |
| $\mu$ | Viscosity of gas | $ML^{-1}T^{-1}$ |
| $\mu_L$ | Viscosity of liquid | $ML^{-1}T^{-1}$ |
| $\rho$ | Density of gas | $ML^{-3}$ |
| $\rho_L$ | Density of liquid | $ML^{-3}$ |
| $\phi$ | Correction factor for concentrated solutions | — |
| *Re.* | Reynolds Number | — |
| *Sc.* | Schmidt Number | — |

Suffixes

| | |
|---|---|
| 1 | denotes conditions at bottom of packed column or at plane 1 |
| 2 | denotes conditions at top of packed column or at plane 2 |
| *e* | denotes equilibrium value |
| *i* | denotes value at interface |
| *G* | denotes gas phase |
| *L* | denotes liquid phase |
| lm | denotes logarithmic mean value |
| *n* | denotes values on plate *n* |

*G*, *OG*, *L*, *OL* refer to gas film, overall gas, liquid film, and overall liquid transfer units

## CHAPTER 20

# Liquid-Liquid Extraction

THE separation of the components of a liquid mixture by treatment with a solvent in which one or more of the desired components is preferentially soluble is known as liquid-liquid extraction. This process has been used in the working up of coal tar liquors, and in recent years it has been very extensively applied to the separation of hydrocarbons in the petroleum industry. Thus, phenol may be extracted from coal tar liquor by the use of benzene, and toluene from petroleum fractions with methanol. The separation of aromatics from paraffins and naphthenic hydrocarbons by use of liquid sulphur dioxide in the Edeleanu process represents a more complex example of this technique. Liquid-liquid extraction is in many ways complementary to distillation, and the two methods will together enable almost all separations to be achieved. Thus, distillation is either uneconomic or impracticable in the following systems.

(1) Where an excessive amount of steam must be used.

(2) Where the relative volatility of the two main components is near unity.

(3) Where azeotropes are formed.

The illustrations given earlier all fall into one or other of these groups. Whilst in distillation we exploit differences in the volatilities of the components, in extraction we make use of variations in their solubilities. A further useful application of the process is exemplified by the recovery of acetic acid by extraction with some solvent such as ethyl acetate, followed by the distillation of the extract obtained. This two-stage process offers a considerable saving in steam, as compared with the direct distillation of a very weak acetic acid. Finally, the method is widely used in the purification of products in the organic chemical industry, the common process of washing with water falling into this class. All such processes require:

(a) Bringing the solvent and solution into intimate contact,

(b) Separation of the resulting two phases, and

(c) Removal and recovery of the solvent from each phase.

## GENERAL CONSIDERATIONS OF PROCESS AND EQUIPMENT

Extraction may be carried out either as a batch or as a continuous process. In the single-stage batch process illustrated in Fig. 20.1, the solvent and solution are mixed together in the mixer and then allowed to separate into the two phases; the "extract" $E$ containing the required solute in the added solvent; and the "raffinate" $R$ the weaker solution with some of the added solvent. With this simple equipment, mixing and separation occur in the same vessel, but a separate vessel may be used for separation. A series of mixers and "settlers" (Fig. 20.2) may be arranged to give a multiple contact system of either the co-current or the countercurrent type.

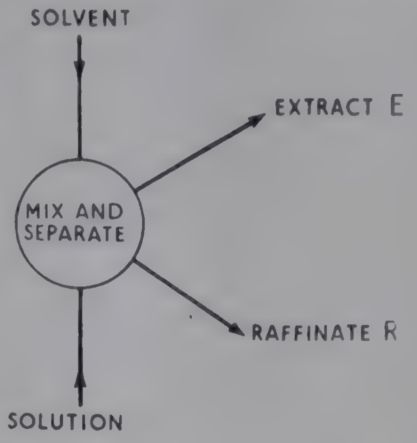

Fig. 20.1. Single-stage batch extraction

Continuous operation is obtained by using either a column apparatus or some form of centrifugal unit. The column apparatus is similar in principle to that discussed in Chapters 18 and 19, but it must be remembered that here there are two liquid phases.

The mechanism of transfer of the solute from the initial solvent to the new solvent is one of diffusion, and the whole process is basically similar to that of absorption, with the gas phase replaced by a second liquid phase. In calculating the capacity of extraction equipment, we are mainly concerned with the equilibrium of the solute between the two solvents, and the resistance to diffusion of the solute. On the basis of the two-film theory, it will be assumed that there are two liquid films, one on each side of the interface.

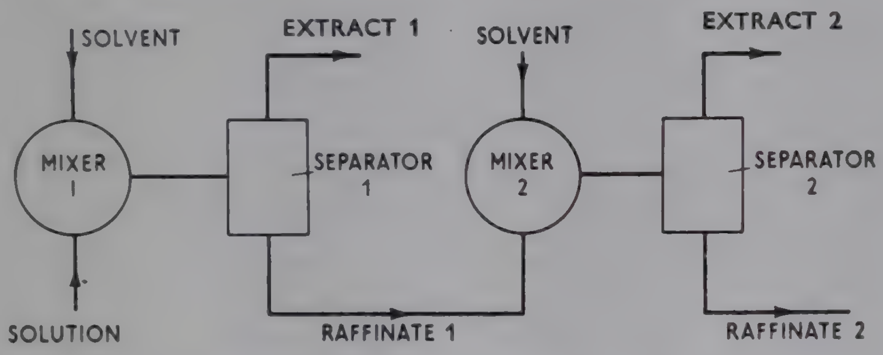

Fig. 20.2. Multiple contact system with fresh solvent

## Equilibrium Conditions

The equilibrium condition for the distribution of one solute between two liquid phases is conveniently considered in terms of the distribution law. Thus, at equilibrium, the ratio of the concentrations of the solute in the two phases is given by $C_1/C_2 = K'$, where $K'$ is the distribution constant. This relation will only apply accurately if both solvents are immiscible, and if there is no association or dissociation of the solute. If the solute forms molecules of different molecular weight, then the distribution law holds for each molecular species. Where the concentrations are small, the distribution law usually holds provided no chemical reaction occurs.

*Ternary Systems.* The addition of a new solvent to a binary mixture of the solute in a solvent may lead to the formation of several types of mixture:

(a) A homogeneous solution may be formed; then the selected solvent is unsuitable.

(b) The solvent may be completely immiscible with the initial solvent.

(c) The solvent may be partially miscible with the original solvent, resulting in the formation of one pair of partially miscible liquids.

(d) The new solvent may lead to the formation of two or three partially miscible liquids.

Of these possibilities, types (b), (c), and (d) all give rise to systems that may be used, but those of types (b) and (c) are the most promising. With conditions of type (b), the equilibrium relation is conveniently shown by a plot of the concentration of solute in one phase against the concentration in the second phase. Conditions given by (c) and (d) are usually represented by triangular diagrams. Equilateral triangles will be used, but it is also possible to employ right-angled isosceles triangles, which have already been discussed in Chapter 17.

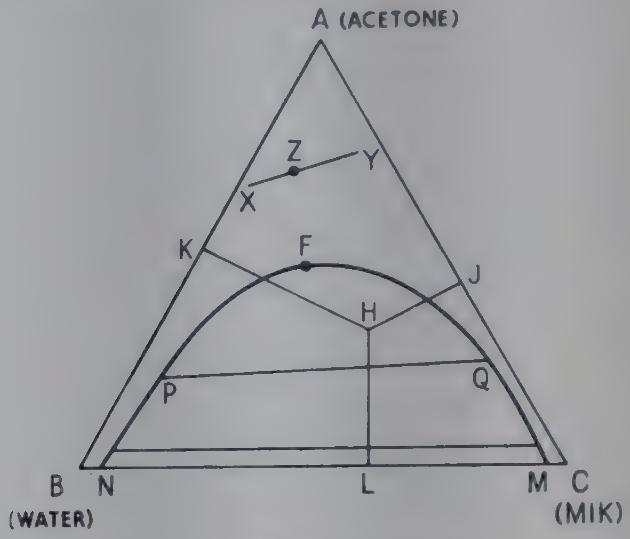

Fig. 20.3. Equilibrium relationship for acetone distributed between water and methyl isobutyl ketone

***Use of Triangular Diagrams.*** The system, acetone($A$) -water($B$) -methyl isobutyl ketone ($C$), as indicated on Fig. 20.3, is of type (c) above. Here the solute $A$ is completely miscible with the two solvents $B$ and $C$, but the two solvents are only partially miscible with each other. Then a mixture indicated by point $H$ consists of the three components $A$, $B$ and $C$ in the ratio of the perpendiculars $HL$, $HJ$, $HK$. The distance $BN$ represents the solubility of solvent $C$ in $B$, and $MC$ that of $B$ in $C$. The area under the curved line $NPFQM$ (the binodal solubility curve) represents a two phase region which will split up into two layers in equilibrium with each other. These layers have compositions represented by points $P$ and $Q$, and $PQ$ is known as a "tie line." Such lines, two of which are shown in the diagram, connect together two phases in equilibrium with each other, and these points must be found by experiment. There will be one point on the binodal curve at $F$ which represents a single phase which does not split into two phases. $F$ is known as a "plait" point, and must also be found by experiment. The plait point is fixed if either the temperature or the pressure is fixed. Inside the area under the curve, the temperature and composition of one phase will fix the composition of the other. For, applying the phase rule to the three-component system at constant temperature and pressure, the number of degrees of freedom is equal to 3 minus the number of phases. In the area where there is only one liquid phase, there are two degrees of freedom and two compositions must be stated. In a system where there are two liquid phases, there is only one degree of freedom.

One of the most useful features of this method of representation is that, if a solution of composition $X$ is mixed with one of composition $Y$, then the resulting mixture will have a composition shown by $Z$ on a line $XY$, such that

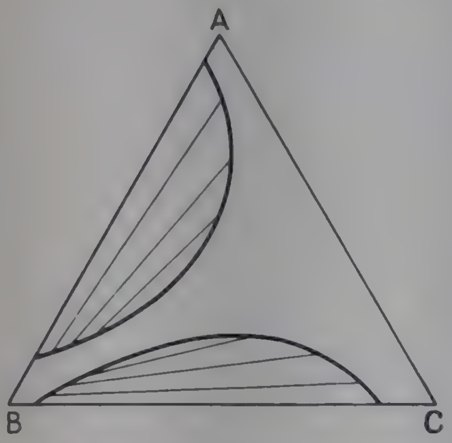

Fig. 20.4. Equilibrium relationship for aniline-water-phenol system

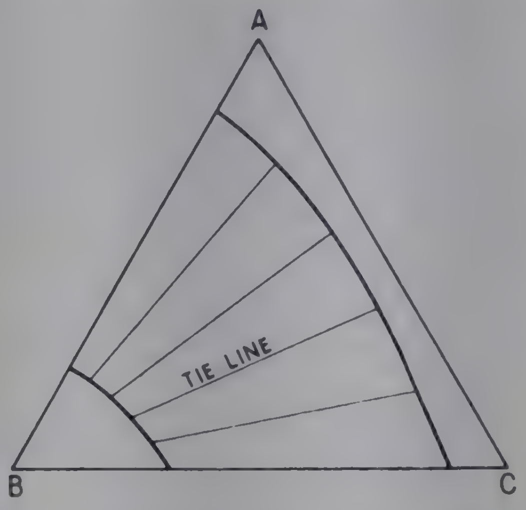

Fig. 20.5. Equilibrium relationship for aniline-water-phenol system

$$\frac{XZ}{ZY} = \frac{\text{amount of } Y}{\text{amount of } X}.$$

Similarly, if from a mixture $Z$ we remove an extract $Y$, the remaining liquor will have composition $X$.

In Fig. 20.4, two separate two-phase regions are formed, whilst in Fig. 20.5, the two-phase regions merge on altering the temperature. Aniline($A$), water($B$), phenol($C$) represents a system of this type. Under the conditions shown in Figs. 20.4 and 20.5, $A$ and $C$ are miscible in all proportions, but $B$ and $A$, and $B$ and $C$ are only partially miscible.

Whilst these diagrams are of considerable use in presenting equilibrium data, Fig. 20.6 is in many ways more useful for determining the selectivity of the solvent, and the number of stages that are likely to be required. In Fig. 20.6, the percentage of solute in one phase is plotted against the percentage in the second phase in equilibrium with it. This is equivalent to plotting the compositions at either end of a tie line. The important factor in assessing the value of a solvent is the ratio of the concentrations of the desired component in the two phases, rather than the actual concentrations. We can thus define a selectivity ratio as

$$\beta = \left[\frac{x_A}{x_B}\right]_E \Big/ \left[\frac{x_A}{x_B}\right]_R \qquad \ldots.(20.1)$$

where $x_A$ and $x_B$ are the mass or mol fractions of $A$ and $B$ in the two phases ($E$) and ($R$). This will be seen to be analogous with the relative volatility in distillation.

## METHOD OF CALCULATING THE NUMBER OF THEORETICAL STAGES REQUIRED

For calculating the number of ideal stages required for a given degree of separation, we make use of the conditions of equilibrium expressed by one of the methods indicated above. We shall consider, in the first place, the number of stages where we have single or multiple contact equipment, and later deal with those conditions where the concentration change is continuous.

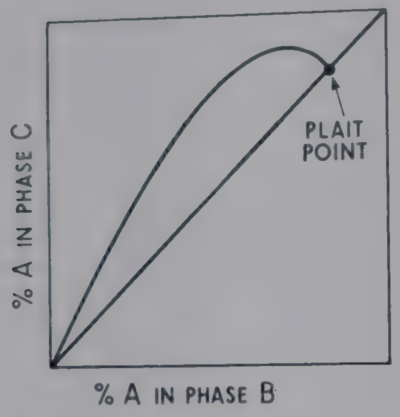

Fig. 20.6. Equilibrium distribution of solute $A$ in phases $B$ and $C$

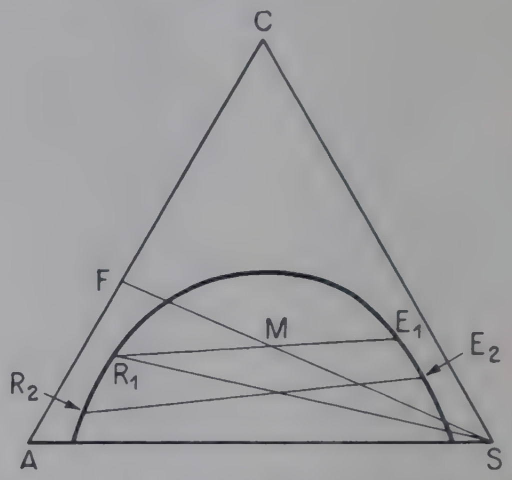

Fig. 20.7. Multiple contact with fresh solvent used at each stage

### Co-current Single and Multiple Contact Arrangements

For the general case where the solvents are partially miscible, the feed solution $F$ is brought into contact with the selective solvent $S$, to give raffinate $R_1$ and an extract $E_1$. The addition of streams $F$ and $S$ is shown on the triangular diagram (Fig. 20.7), by the point $M$, where $\frac{FM}{MS} = \frac{S}{F}$. This mixture $M$ breaks down to give extract $E_1$ and raffinate $R_1$, at opposite ends of a tie line through $M$.

If a second stage is used, then the raffinate $R_1$ is treated with a further quantity of solvent $S$, and extract $E_2$ and raffinate $R_2$ are obtained as shown in the figure.

***Solvent recovery.*** The complete process consists in carrying out the extraction, and recovering the solvent from the raffinate and extract obtained. Thus, for a single stage system as indicated in Fig. 20.8, the raffinate $R$ is passed into the distillation column where it is separated to give purified raffinate $R'$ and solvent $S_R$. The extract $E$ is passed to another distillation unit to give extract $E'$ and a solvent stream $S_E$. These recovered solvents $S_R$ and $S_E$ are pumped back to the extraction process as shown. We can represent this cycle on the diagram (Fig. 20.9), by showing the removal of $S_R$ from $R$ to give composition $R'$, and the removal of $S_E$ from $E$ to give composition $E'$. It has been assumed in this case that perfect separation is obtained in the stills, so that pure solvent is obtained in the streams $S_R$ and $S_E$, but the same form of diagram can be used where imperfect separation is obtained. It should be noted that, when $ES$ is a tangent to the binodal curve, we have the maximum concentration of solute $C$ in the extract $E'$. It also follows that $E'$ represents the maximum possible concentration of $C$ in the feed. Sufficient solvent $S$ must be used to bring the mixture $M$ within the two-phase area.

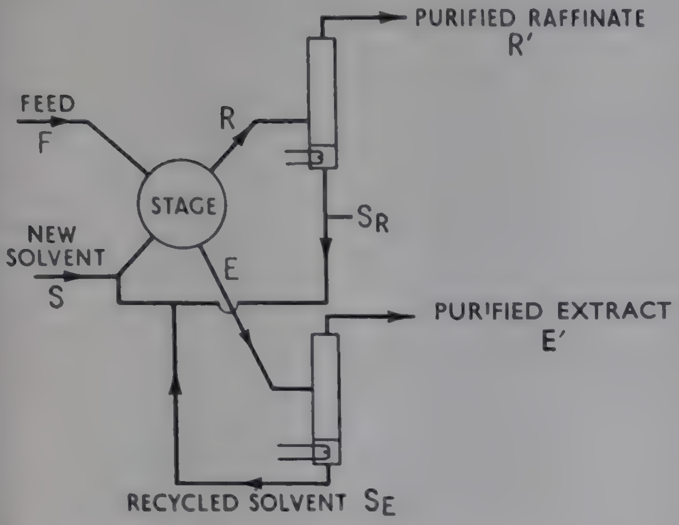
PURIFIED RAFFINATE
R′
FEED
F
R
STAGE
NEW SOLVENT
S
E
S_R
PURIFIED EXTRACT
E′
RECYCLED SOLVENT S_E

Fig. 20.8. Single-stage process with solvent recovery

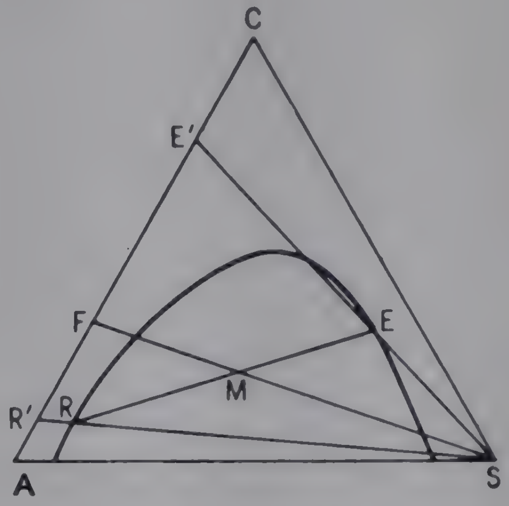

Fig. 20.9. Representation of process shown in Fig. 20.8

***Co-current Multiple Contact with Immiscible Solvents.*** In this case, as illustrated in Fig. 20.10, there is no need to use triangular diagrams. Suppose

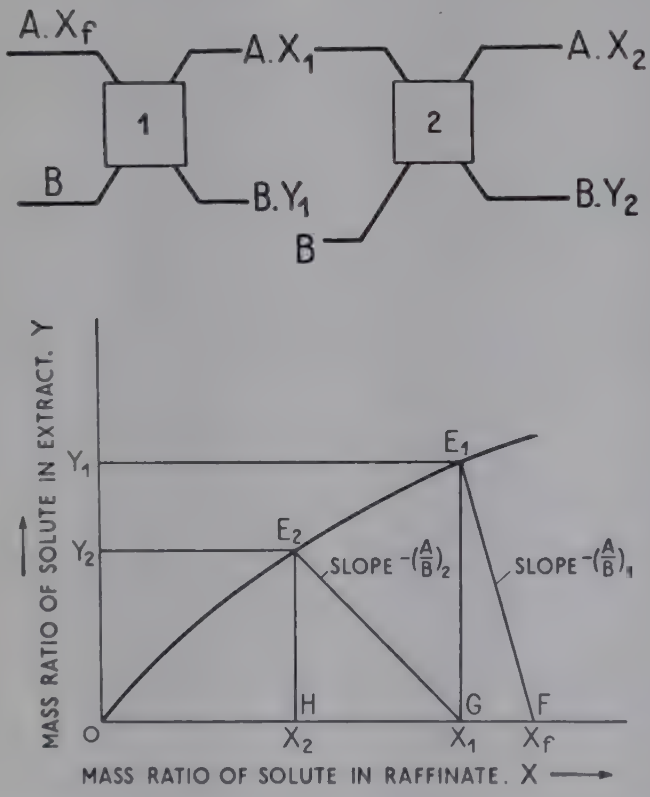
A.X_f
A.X_1
A.X_2
1
2
B
B.Y_1
B
B.Y_2
MASS RATIO OF SOLUTE IN EXTRACT. Y
Y_1
Y_2
E_1
E_2
SLOPE −(A/B)_2
SLOPE −(A/B)_1
O
H
G
F
X_2
X_1
X_f
MASS RATIO OF SOLUTE IN RAFFINATE. X

Fig. 20.10. Calculation of number of stages for a co-current multiple-contact process, using immiscible solvents

the initial solution contains $A$ lbs of solvent $A$ with a mass ratio $X_f$ of solute. Then the selective solvent to be added will be $B$ lbs of solvent $B$. On mixing and separating we shall get a raffinate with the solvent $A$ containing a mass ratio $X_1$

of solute, and an extract $B$ with a mass ratio $Y_1$ of solute. A material balance on the solute gives,

$$AX_f = AX_1 + BY_1$$

$$\therefore \qquad \frac{Y_1}{X_1 - X_f} = -A/B. \qquad \ldots.(20.2)$$

We can illustrate this process by letting the point $F$ represent the feed solution and drawing a line $FE_1$, of slope $-\left(\frac{A}{B}\right)_1$, to cut the equilibrium curve in $E_1$. This will then give the composition $Y_1$ of the extract and $X_1$ of the raffinate. If a further stage is then carried out by the addition of solvent $B$ to the stream $AX_1$, then point $E_2$ is found on the equilibrium curve by drawing $GE_2$ of slope $-\left(\frac{A}{B}\right)_2$. $E_2$ then gives the compositions $X_2$ and $Y_2$ of the final extract and raffinate. This system can be used for any number of stages, with any assumed variation in the proportion of solvent $B$ to raffinate from stage to stage.

*Conditions where the Distribution Law is followed.* If the distribution law is followed, then the equilibrium curve becomes a straight line of equation, $Y = mX$. We can then rewrite the material balance on the solute as below.

$$AX_f = AX_1 + BY_1 = AX_1 + BmX_1 = (A + Bm)X_1$$

$$\therefore \qquad X_1 = \left[\frac{A}{A + Bm}\right] . X_f. \qquad \ldots.(20.3)$$

If a further $B$ lbs of $B$ is added to raffinate $AX_1$ to give extract of composition $Y_2$ and raffinate $X_2$ in a second stage, we get;

$$AX_1 = AX_2 + BmX_2 = X_2\,[A + Bm]$$

$$\therefore \qquad X_2 = \left[\frac{A}{A + Bm}\right] X_1 = \left[\frac{A}{A + Bm}\right]^2 . X_f \qquad \ldots.(20.4)$$

And for $n$ stages

$$X_n = \left[\frac{A}{A + Bm}\right]^n . X_f \qquad \ldots.(20.5)$$

or the number of stages

$$n = \frac{\log X_n/X_f}{\log \left[\frac{A}{A + Bm}\right]} \qquad \ldots.(20.6)$$

## Countercurrent Multiple Contact with Immiscible Solvents

If we arrange a series of mixing and separating vessels so that the flow is countercurrent, then the conditions of flow can be represented as in Fig. 20.11, where each circle corresponds to a mixer and a separator. The initial solution $F$ of the solute $B$ in solvent $A$ is fed to the first unit and leaves as raffinate $R_1$. This stream passes through the units and leaves from the $n$th unit as stream $R_n$.

The fresh solvent $S$ enters at the $n$th unit and passes in the reverse direction through the units, leaving as extract $E_1$.

Let $X$ denote the ratio of solute to solvent in the raffinate streams, and $Y$ denote the ratio of the solute to solvent in the extract streams.

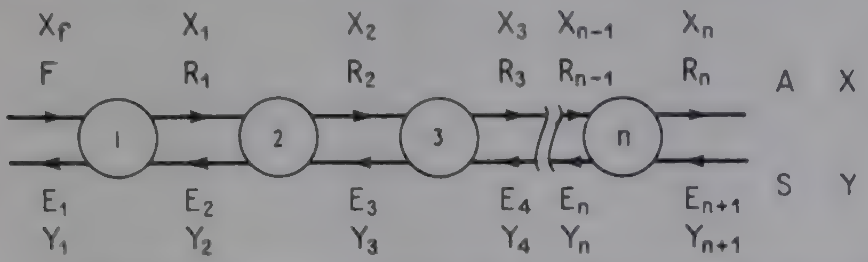

Fig. 20.11. Arrangement for multiple-contact extraction in countercurrent flow

Since the two solvents are immiscible, the solvent in the raffinate streams remains as $A$ units, and the added solvent in the extract streams as $S$. We can then write down the following material balances for the solute:

(1) For 1st stage

$$AX_f + SY_2 = AX_1 + SY_1$$

(2) For $n$th stage

$$AX_{n-1} + SY_{n+1} = AX_n + SY_n$$

(3) For the whole unit

$$AX_f + SY_{n+1} = AX_n + SY_1$$

$$\therefore \quad Y_{n+1} = \frac{A}{S}(X_n - X_f) + Y_1 \qquad \ldots.(20.7)$$

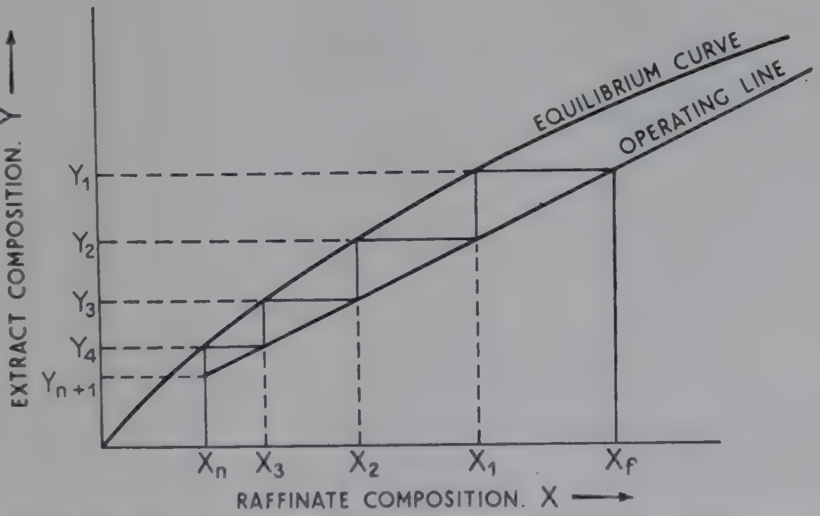

Fig. 20.12. Graphical method for determining the number of stages for process shown in Fig. 20.11, using immiscible solvents

This is the equation of a straight line of slope $\frac{A}{S}$, known as the operating line; from the above equalities, it is seen to pass through the points $(X_f, Y_1)$ and $(X_n, Y_{n+1})$. In Fig. 20.12 is drawn the equilibrium relation ($Y_n$ v $X_n$) and the operating line, and the number of stages required to pass from $X_f$ to $X_n$ is found by drawing in steps between the operating line and the equilibrium curve: in the example shown four stages are required, and $(X_n, Y_{n+1})$ correspond to $(X_4, Y_5)$. It should be noted that the operating line connects the composition of the raffinate stream leaving $(X_n)$ and the fresh solvent stream entering a unit $(Y_{n+1})$.

### Countercurrent Stagewise Process with Partially Miscible Solvents

The arrangement of the equipment will be the same as for the previous case with immiscible solvents but, since the amount of solvent in the extract and raffinate streams is changing, the material balance is taken for the total streams entering and leaving each stage.

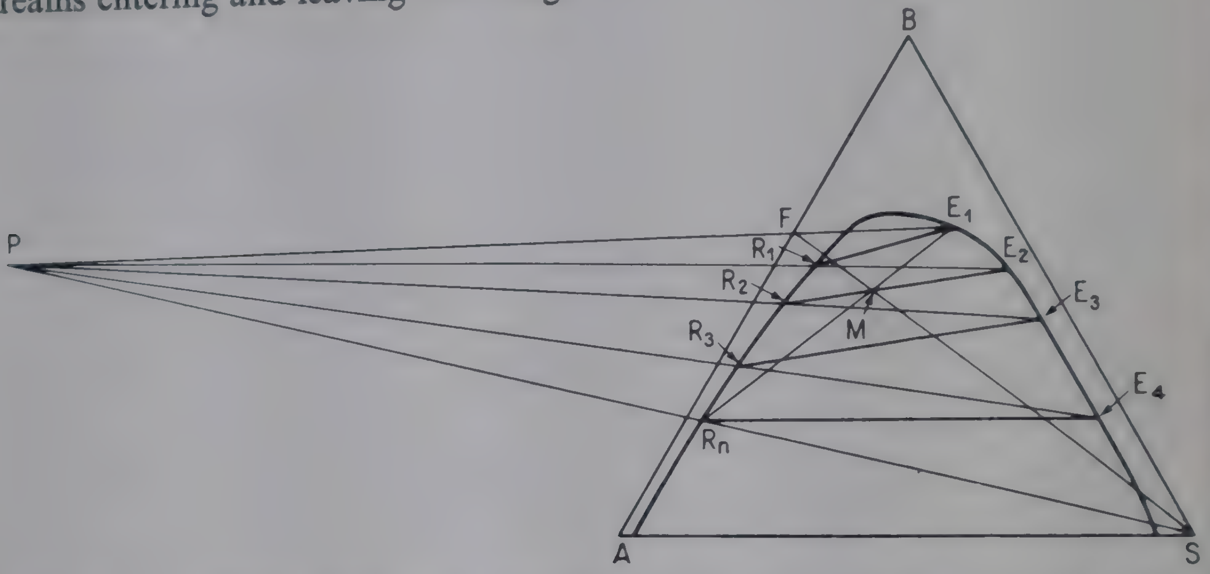

Fig. 20.13. Graphical method for determining the number of stages for process shown in Fig. 20.11, using partially miscible solvents

With the notation as before (Fig. 20.11), suppose that the feed $F$, final extract $E_1$, fresh solvent $S (= \text{stream } E_{n+1})$ and final raffinate $R_n$ to be fixed. Then taking material balances:

(1) *Over 1st Unit*

$$F + E_2 = R_1 + E_1$$

$$\therefore \quad F - E_1 = R_1 - E_2 = P, \text{ say—the difference stream} \quad \ldots.(20.8)$$

(2) *Over stages 1 — n*

$$F + E_{n+1} = R_n + E_1 = M, \text{ say} \quad \ldots.(20.9)$$

$$\therefore \quad F - E_1 = R_n - E_{n+1} = P. \quad \ldots.(20.10)$$

(3) *Over nth Unit.*

$$R_{n-1} + E_{n+1} = E_n + R_n$$

$$\therefore \quad R_{n-1} - E_n = R_n - E_{n+1} = P \quad \ldots.(20.11)$$

Thus the difference in quantity between the raffinate leaving a stage $R_n$, and the extract entering from next state $E_{n+1}$, is constant. Equation 20.11 will also apply to each of the solvents and the solute, and therefore the difference stream will have a constant composition (cf. equation 17.38). This means that, with the notation of a triangular diagram, lines joining any two points representing $R_n$ and $E_{n+1}$ will pass through a common pole. The number of stages required to pass from an initial concentration $F$ to a final raffinate concentration $R_n$ can then be found using a triangular diagram, by the method shown in Fig. 20.13.

Join the points $F$ and $S$ representing the compositions of the feed and fresh solvent $S$. Then the composition of a mixture of $F$ and $S$ is shown by point $M$ where

$$\frac{MS}{MF} = \frac{\text{mass of } F}{\text{mass of } S}.$$

Draw a line from $R_n$ through $M$ to give $E_1$ on the binodal curve. Draw $E_1 F$ and $SR_n$ to meet at the pole $P$. It should be noted that $P$ represents an imaginary mixture, as already found for the leaching problems discussed in Chapter 17.

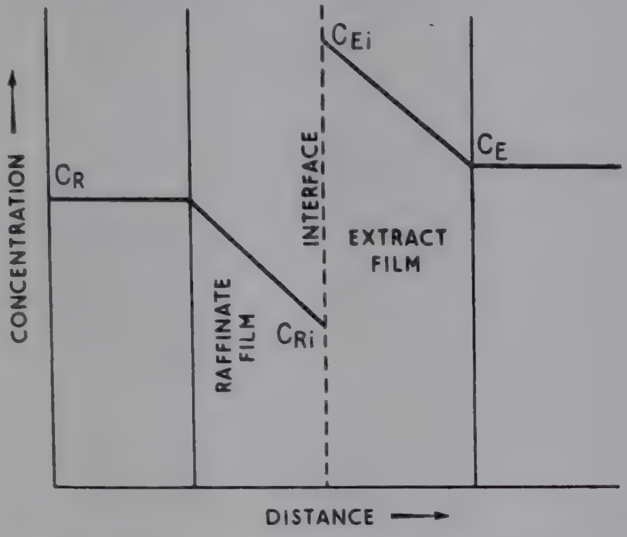

Fig. 20.14. Concentration profile near an interface

Then, in an ideal stage, the extract $E_1$ leaves in equilibrium with the raffinate $R_1$, so that the point $R_1$ is at the end of the tie line through $E_1$. To find the extract $E_2$, draw $PR_1$ to cut the binodal curve at $E_2$. Then the points $R_2, E_3, R_3, E_4$, etc., can be found in the same way. If the final tie line (say, $E_4R_4$) does not pass through $R_n$, then the amount of solvent added is incorrect for the desired change in composition. This does not generally invalidate the method, since it will give the required number of ideal stages sufficiently accurately.

## Continuous Extraction in Column Apparatus

The use of spray or packed towers enables continuous countercurrent extraction to be obtained in a similar manner to gas absorption or distillation. Applying the two-film theory of diffusion, the concentration gradients for transfer of a desired solute from a raffinate to an extract phase are as shown in Fig. 20.14, which is similar to Fig. 19.1 for gas absorption.

The transfer through the film on the raffinate side of the interface is brought about by a concentration difference $(C_R - C_{Ri})$, and through the film on the extract side by a concentration difference $C_{Ei} - C_E$.

The rate of transfer across these films can be expressed, as in absorption, in the following way:

$$N = k_R \,.\, (C_R - C_{Ri}) = k_E(C_{Ei} - C_E) \qquad \ldots.(20.12)$$

where $C_R$, $C_E$ are the concentrations of the solute in the raffinate and extract phases in mols per unit volume,

$k_R$, $k_E$ are the transfer coefficients for raffinate and extract films, respectively, and

$N$ is the molar rate of transfer per unit area.

Then
$$\frac{k_R}{k_E} = \frac{C_{Ei} - C_E}{C_R - C_{Ri}} = \frac{\Delta C_E}{\Delta C_R} \qquad \text{....(20.13)}$$

If the equilibrium curve can be taken as a straight line, then assuming equilibrium at the interface,

$$C_{Ei} = mC_{Ri} \qquad \text{....(20.14a)}$$

Further,
$$C_E = mC_R^* \qquad \text{....(20.14b)}$$

and
$$C_E^* = mC_R \qquad \text{....(20.14c)}$$

where $C_E^*$ is the concentration in phase $E$ in equilibrium with $C_R$ in phase $R$, and $C_R^*$ is the concentration in phase $R$ in equilibrium with $C_E$ in phase $E$.

We can also write the relations for mass transfer in terms of overall transfer coefficients $K_R$ and $K_E$ defined by the relation,

$$N = K_R(C_R - C_R^*) = K_E(C_E^* - C_E) \qquad \text{....(20.15)}$$

and by similar reasoning to that for absorption (Chapter 19),

$$\frac{1}{K_R} = \frac{1}{k_R} + \frac{1}{mk_E} \qquad \text{....(20.16)}$$

and
$$\frac{1}{K_E} = \frac{1}{k_E} + \frac{m}{k_R} \qquad \text{....(20.17)}$$

***Capacity of Column operating as Continuous Countercurrent Unit.*** The capacity of a column operating as a countercurrent extractor, as indicated in Fig. 20.15, can be derived as below.

If $L_R$, $L_E$ are the flow rates of raffinate and extract phases in volumes per unit area,

$a$ is the interfacial surface per unit volume, and

$Z$ is the height of packing,

then, over a small height d$Z$, a material balance gives

$$L_R \,.\, \mathrm{d}C_R = L_E \,\mathrm{d}C_E \qquad \text{....(20.18)}$$

From equation 20.12,

$$L_R \,.\, \mathrm{d}C_R = k_R(C_R - C_{Ri})a \,\mathrm{d}Z$$

$$\therefore \qquad \int_{C_{R2}}^{C_{R1}} \frac{\mathrm{d}C_R}{C_R - C_{Ri}} = \frac{k_R \,.\, a}{L_R} \,.\, Z \qquad \text{....(20.19)}$$

The integral on the left-hand side of this equation is known as the number of raffinate film transfer units $\mathbf{N}_R$, and the height of this transfer unit is

$$\mathbf{H}_R = \frac{L_R}{k_R \,.\, a} \qquad \text{....(20.20)}$$

In a similar manner and by analogy with absorption, we get:

$$\mathbf{H}_E = \frac{L_E}{k_E \,.\, a} = \text{ height of extract film transfer unit} \quad \ldots.(20.21)$$

$$\mathbf{H}_{OR} = \frac{L_R}{K_R \,.\, a} = \text{ height of overall transfer unit based on concentration in raffinate phase.} \quad \ldots.(20.22)$$

$$\mathbf{H}_{OE} = \frac{L_E}{K_E \,.\, a} = \text{ height of overall transfer unit, based on concentration in extract phase.} \quad \ldots.(20.23)$$

Since

$$\frac{1}{K_R} = \frac{1}{k_R} + \frac{1}{mk_E} \quad \ldots.(20.16)$$

$$\frac{L_R}{K_R} = \frac{L_R}{k_R} + \frac{L_R}{mk_E} \times \frac{L_E}{L_E}$$

$\therefore$

$$\mathbf{H}_{OR} = \mathbf{H}_R + \frac{L_R}{mL_E} \,.\, \mathbf{H}_E \quad \ldots.(20.24)$$

and

$$\mathbf{H}_{OE} = \mathbf{H}_E + \frac{mL_E}{L_R} \,.\, \mathbf{H}_R \quad \ldots.(20.25)$$

This is the same form of relation as already obtained for absorption, but it is only with dilute solutions that the group $\frac{mL_E}{L_R}$ is constant.

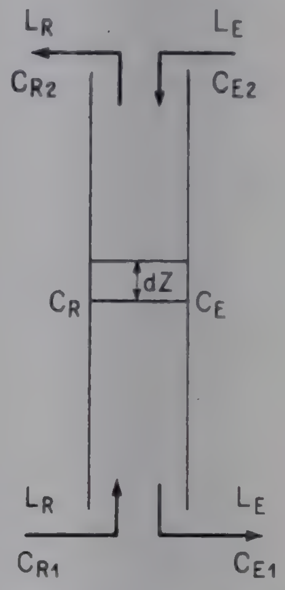

Fig. 20.15. Counter-current flow in a packed column

Leibson and Beckmann[(20)] have presented a plot of this equation as a method of correlating their experimental results (Fig. 20.16).

In deriving these relationships, it has been assumed that $L_R$ and $L_E$ are constant throughout the tower; this will not be true if a large part of the solute is transferred from a concentrated solution to the other phase. Again, it is assumed that the transfer coefficients are independent of concentration. For dilute solutions and where the equilibrium relation is a straight line, a simple expression can be obtained for determining the required height of a column, by the same method as in Chapter 19.

Thus $L_R \,.\, \mathrm{d}C_R = K_R(C_R - C_R^*) \,.\, a\,\mathrm{d}Z$ can be integrated over the height $Z$ and written as

$$L_R \,.\, [C_{R1} - C_{R2}] = K_R(\Delta C_R)_{\mathrm{lm}} a \,.\, Z \quad \ldots.(20.26)$$

where $(\Delta C_R)_{\mathrm{lm}}$ is the logarithmic mean of $(C_R - C_R^*)_1$ and $(C_R - C_R^*)_2$. This simple relation has been used by workers in the determination of $K_R$ or $K_E$ in small laboratory columns; care should be taken when applying many of these results to other conditions.

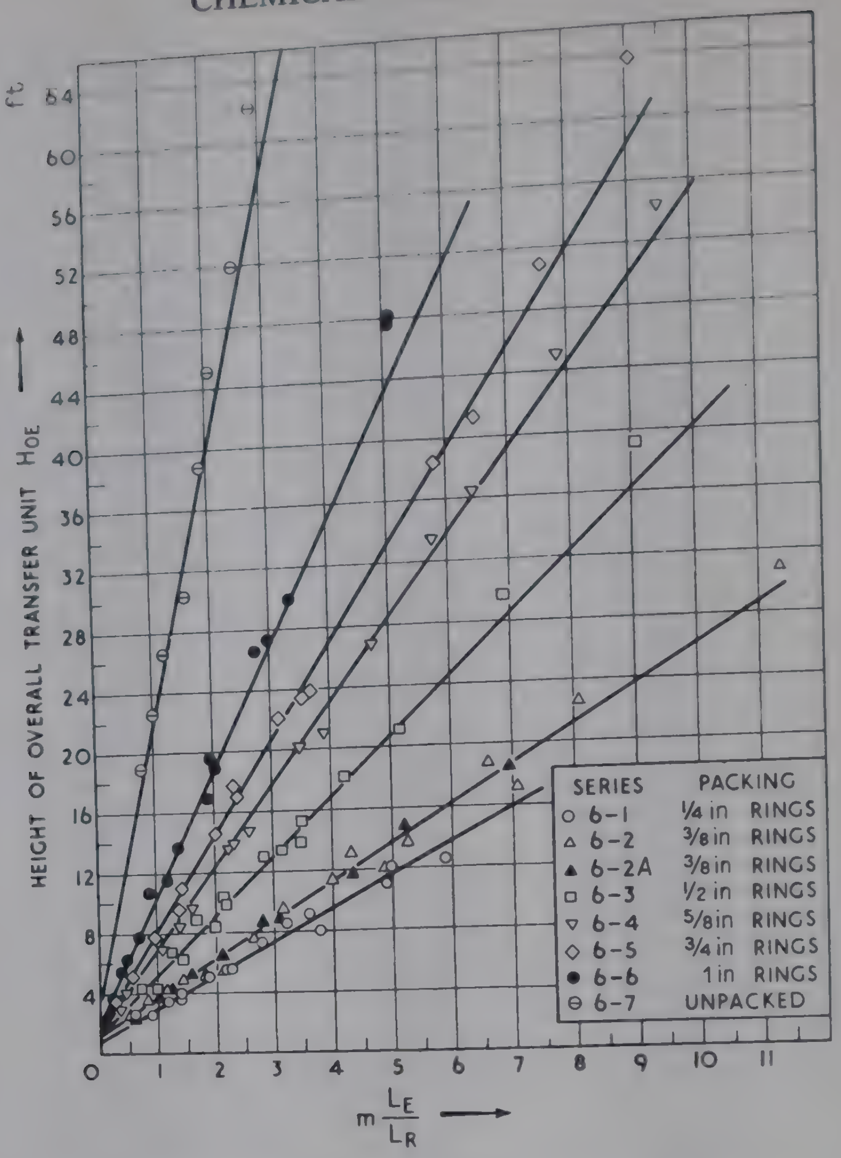

Fig. 20.16. Height of the transfer unit $\mathbf{H}_{OE}$ as a function of $\frac{mL_E}{L_R}$ for various packings.

Transfer of diethylamine from water to dispersed toluene

**Example.** In the extraction of acetic acid from an aqueous solution with benzene in a packed column of height 4·5 feet and cross-sectional area 0·049 ft², the concentrations measured at the inlet and outlet of the column are as shown in Fig. 20.17.

| | |
|---|---|
| Acid concentration in inlet water phase $C_{W2}$ | $= 0{\cdot}0430$ lb-mol/ft³ |
| Acid concentration in outlet water phase $C_{W1}$ | $= 0{\cdot}0427$ lb-mol/ft³ |
| Flow rate of benzene phase | $= 0{\cdot}72$ ft³/hr |
| | $= 14{\cdot}6$ ft³/ft²-hr |
| Inlet benzene phase concentration $C_{B1}$ | $= 0{\cdot}000248$ lb-mol/ft³ |
| Outlet benzene phase concentration $C_{B2}$ | $= 0{\cdot}000716$ lb-mol/ft³ |

Determine the overall transfer coefficient and the height of the transfer unit.

**Solution.** The acid transferred to the benzene phase is

$$0{\cdot}72(0{\cdot}000716 - 0{\cdot}000248) = 0{\cdot}000337 \text{ lb-mol/hr}$$

The equilibrium relationship for this system is $\left[\frac{C_B}{C_W}\right]$ at equilibrium $= 0{\cdot}0247$.

$$\therefore \qquad C^*_{B1} = 0{\cdot}0247 \times 0{\cdot}0427 = 0{\cdot}001055$$

and

$$C^*_{B2} = 0{\cdot}0247 \times 0{\cdot}0430 = 0{\cdot}001062$$

$$\therefore \qquad \text{Driving force at bottom } \Delta C_1 = 0{\cdot}001055 - 0{\cdot}000248 = 0{\cdot}000807$$

and Driving force at top $\Delta C_2 = 0{\cdot}001062 - 0{\cdot}000716 = 0{\cdot}000346$

$\therefore$ Log mean driving force $= \Delta C_{lm} = 0{\cdot}00544$

$$\therefore \quad K_B\,.\,a = \frac{0{\cdot}000337}{0{\cdot}2214 \times 0{\cdot}000544} = \frac{\text{mols transferred}}{\text{volume of packing} \times \Delta C_{lm}}$$

$$= \underline{\underline{2{\cdot}80 \text{ lb-mol/hr-ft}^3 \text{ (lb-mol/ft}^3)}}$$

and
$$\mathbf{H}_{OB} = \frac{14{\cdot}6}{2{\cdot}80} = \underline{\underline{5{\cdot}25 \text{ ft.}}}$$

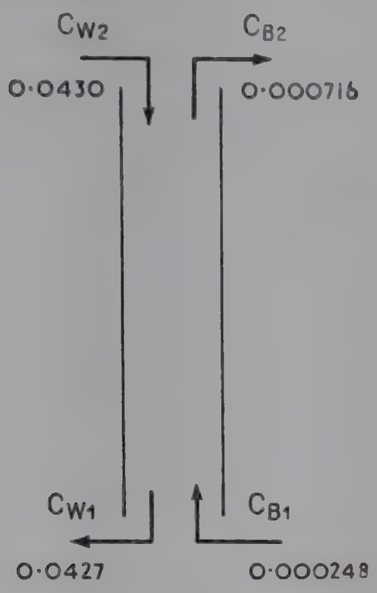

Fig. 20.17.

## VALUES OF TRANSFER COEFFICIENTS AND HEIGHTS OF TRANSFER UNITS

A number of workers have measured the values of transfer coefficients or the heights of transfer units in small laboratory columns, and correlated their results by methods similar to those used in absorption. Although it may be assumed that the transfer coefficients are related to the Reynolds and Schmidt groups, no simple relationship has been proposed that has proved entirely satisfactory. The problem here is complicated by the fact that in extraction both phases may offer an appreciable resistance to transfer, and it is necessary to include the Reynolds group for both the continuous and the dispersed phase.

A correlation of the form of equations 20.24 and 20.25 is not usually found reliable; $\mathbf{H}_D$ for the dispersed phase is nearly independent of the flow rates of either phase, whilst $\mathbf{H}_C$ for the continuous phase depends on the flow rates of both phases. On this basis

$$\mathbf{H}_C = \phi \left[\frac{u_C}{u_D}\right]^s \qquad \ldots.(20.27)$$

where $u_C$ and $u_D$ are the volumetric flow rates per unit area of the continuous and dispersed phases. DARK[(23)], working on the system acetic acid-benzene-water, was able to express his results in the form

$$\frac{1}{K_D\,.\,a} = \frac{4{\cdot}67}{u_D} + 0{\cdot}0032 u_C{}^{0{\cdot}84} \qquad \ldots.(20.28)$$

which suggests that $\mathbf{H}_D$ for the dispersed phase is almost independent of the flow rate $u_C$ of the continuous phase. Here $u_C$ and $u_D$ are in ft/hr and $K_D$ is in ft/hr.

It must be remembered that, with two liquid phases, an increase in the flow downwards of the continuous phase will increase the hold-up, and hence the effective interfacial area. Thus, with the benzene phase dispersed and at a fixed rate of 21 ft/hr, increase in the continuous water phase gave results as in Fig. 20.18, the individual curves corresponding to different heights of column.

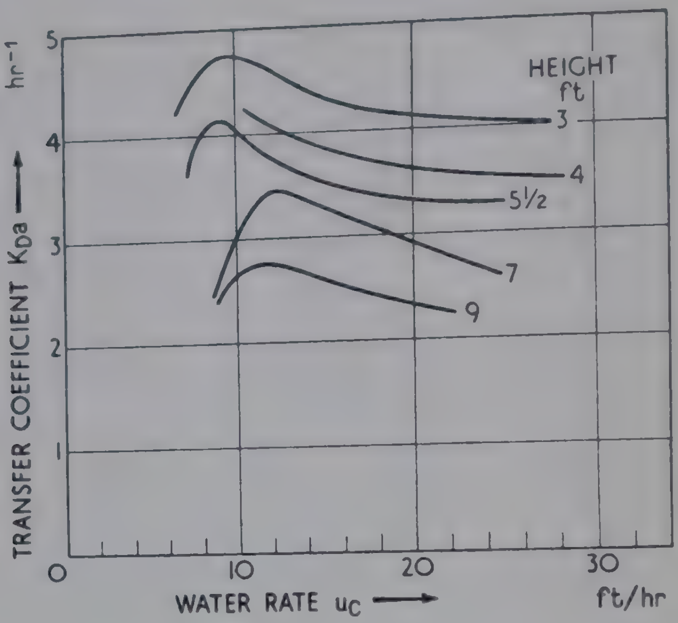

Fig. 20.18. Effect of flow rate of continuous phase (water) on transfer coefficient $K_Da$ for transfer of acetic acid from benzene to water

The eventual falling off of $K_Da$, with an increase of $u_C$ beyond about 10, is believed to be due to the coalescence of the dispersed drops, and the consequent reduction in the surface.

Some results by PRATT and GLOVER(11), for the removal of acetone and acetaldehyde from vinyl acetate with water, are correlated by the equations:

(a) for acetone extracted, and vinyl acetate as dispersed phase:

$$\frac{1}{K_Da} = \frac{2{\cdot}82}{u_D} + \frac{1{\cdot}20}{u_C^{0{\cdot}75}} \qquad \ldots.(20.29)$$

(b) for acetaldehyde extracted, and vinyl acetate as dispersed phase

$$\frac{1}{K_Da} = \frac{3{\cdot}10}{u_D} + \frac{0{\cdot}92}{u_C^{0{\cdot}75}} \qquad \ldots.(20.30)$$

where $K_D \,.\, a$ is the overall transfer coefficient based on the dispersed phase [lb-mols/hr-ft$^3$ (lb-mols/ft$^3$)], and $u_D$, $u_C$ are the flow rates of the dispersed and continuous phases in ft/hr.

Some values of other workers are given in Table 20.1, below:

TABLE 20.1. *Miscellaneous packed-tower extraction data*

| Dispersed phase | Continuous phase | Extracted solute | Direction of extraction | Phase which wets packing | Packing | Tower diam., in. | Distributor nozzles | Temp., °C. | $u_C$ | $u_D$ | Equation $Ka$ in hr$^{-1}$ H in ft |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | Ft/hr | | |
| Water | Toluene | Benzoic acid | $C \rightarrow D$ | $D$ | ½-in. saddles | 2·03 | Sprayco-5B | 15–18 | 22·2 | 13·3–44·4 | $K_Da = 6 + 0\cdot18u_D$ |
| | | | | | | | | | 44·4 | 13·3–44·4 | $K_Da = 7\cdot5 + 0\cdot18u_D$ |
| Water | Benzene | Benzoic acid | $C \rightarrow D$ | $D$ | ½-in. saddles | 1·89 | ................ | 30 | 7·4–96 | 19·2–77·5 | $K_Da = 4\cdot42$ to $12\cdot7$ |
| Toluene | Water | Furfural | $C \rightarrow D$ | .. | ½-in. saddles | 4·0 | ................ | 25 | 14·7–58·0 | 8·30–28·5 | $\mathbf{H}_{OC} = 3\cdot3\left(\frac{u_C}{u_D}\right)^{0\cdot95}$ |
| Toluene | Water | Diethyl amine | $C \rightarrow D$ | .. | 4-mm. glass beads | 1·312 | 2-mm. diam.-tubes | 26·8 | 1·33–8·91 | 0·17–5·20 | $\mathbf{H}_{OC} = 1\cdot73\,\frac{u_C}{mu_D}$ |
| | | | | | | | | 38·5 | 1·68–14·12 | 0·08–8·03 | $\mathbf{H}_{OC} = 1\cdot55\,\frac{u_C}{mu_D}$ |
| | | | | | | | | 48·5 | 2·50–19·20 | 0·28–9·28 | $\mathbf{H}_{OC} = 1\cdot27\,\frac{u_C}{mu_D}$ |
| | | | | | | | | 57·5 | 2·44–16·66 | 0·88–4·64 | $\mathbf{H}_{OC} = 0\cdot79\,\frac{u_C}{mu_D}$ |
| Methyl isobutyl ketone | Water | Acetic acid | $C \rightarrow D$ | $C$ | ½-in. $C$ rings | 3·55 | Six 0·12 in. I.D. nozzles | 23–27 | 10–80 | 30–70 | $\mathbf{H}_{OD} = 0\cdot77\left(\frac{u_D}{u_C}\right)^{0\cdot648}$ |
| | | | | | | | | | 10–60 | 10 | $K_Da = 7\cdot8 + 0\cdot169u_C$ |
| Methyl isobutyl ketone | Water | Acetic acid | $C \rightarrow D$ | $C$ | ½-in. saddles | 3·55 | Six 0·12 in. I.D. nozzles | 23–27 | 10–40 | 40 | $K_Da = 2\cdot13u_C$ |
| Methyl isobutyl ketone | Water | Acetic acid | $C \rightarrow D$ | $C$ | 1-in. $C$ rings | 3·55 | Six 0·12 in. I.D. nozzles | 23–27 | 10–40 | 40 | $K_Da = 15 + 0\cdot64u_C$ |
| Methyl ethyl ketone | $CaCl_2$ brine | Water | $D \rightarrow C$ | $C$ | ½-in. saddles, ½-in. ceramic rings | 3·55 | Six 0·12 in. I.D. nozzles | 25–28 | 16·1–34·1 | 26·1–69·5 | $K_Da = 0\cdot74u_D$ |
| $CaCl_2$ brine | Methyl ethyl ketone | Water | $C \rightarrow D$ | $D$ | ½-in. saddles | 3·55 | Six 0·12 in. I.D. nozzles | 25–28 | 28·0–65·7 | 17·0–17·3 | $K_Ca = 3\cdot8{u_C}^{0\cdot42}$ |

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LEIBSON and BECKMAN(20) have measured the values of coefficients for the transfer of diethylamine from a continuous water phase to dispersed toluene, in columns of 3, 4 and 6 in. diameter, packed with Raschig rings varying from ¼ to 1 in. They found that the size of the drop was independent of the size of packing for rings greater than ½ in., and that the biggest drops were obtained with rings of ¼ in. These results are in reasonable agreement with those of GAYLER and PRATT(17) who found that the hold-up of the dispersed phase decreases as the packing size is increased. On this basis, we should expect poorer results with packings larger than ½ in., since the interfacial area will be reduced. It is not possible to give any simple relation between the transfer coefficient, the diameter of the tower, and the diameter of the packing, but it is clear that transferring from one size of unit to another does lead to some degree of uncertainty.

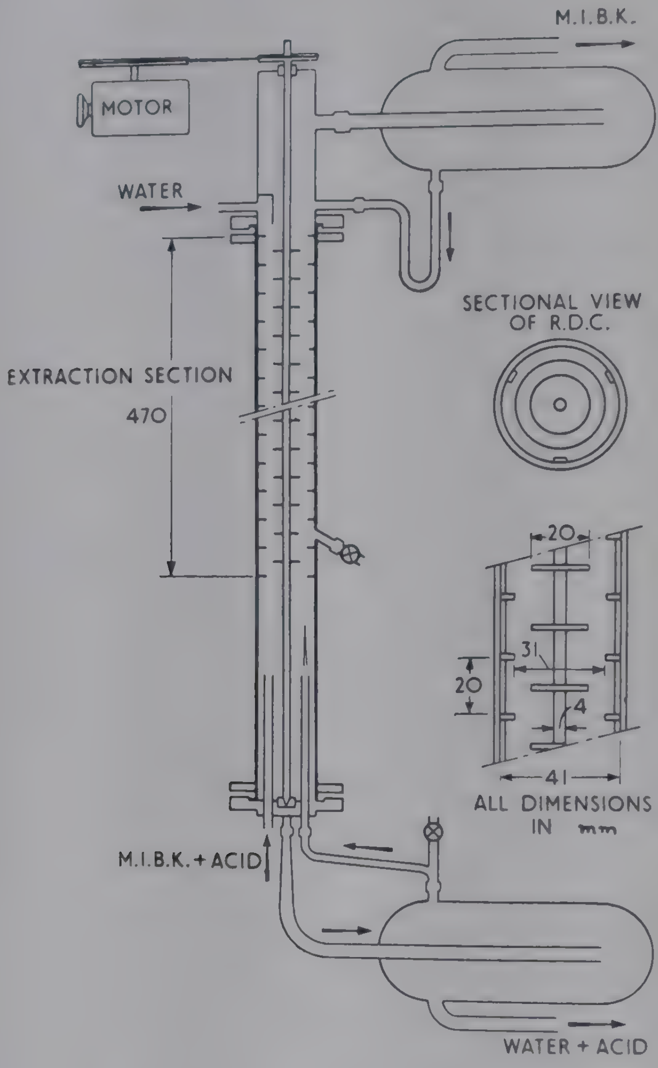

Fig. 20.19. Rotating disc extractor

In order to determine the values of film transfer coefficients, COLBURN and WELSH(10) devised a technique of using two partially miscible liquids and measuring the degree of mixing achieved in their passage through the apparatus. In this way, they eliminated one of the film resistances and were then able to obtain a value for the other. They found, as indicated earlier, that on the whole the height of the transfer unit for the dispersed phase $\mathbf{H}_D$ was independent of the ratio of the flow rates of the two phases, but that $\mathbf{H}_C$ for the continuous phase varied widely with the flow rates. LADDHA and SMITH(12), using the same technique, have found somewhat similar results.

APPEL and ELGIN(6), ROW, KOFFOLT and WITHROW(8), and others have given some data for extraction in small spray towers of from 2 to 9 in. diameter, but again no general correlation has been obtained. Table 20.2 gives some results as presented in the literature from various workers.

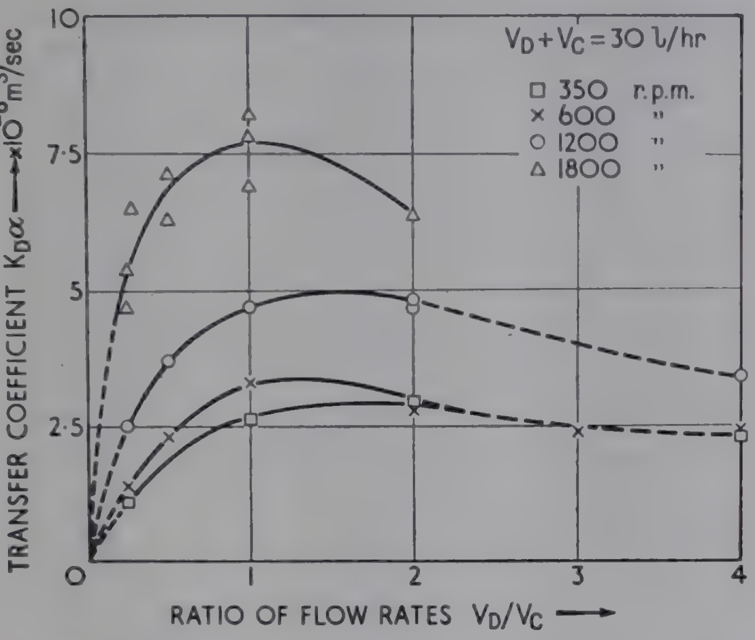

Fig. 20.20. Effect of flow rates on transfer coefficient $K_D\alpha$ in disc extractor

*Rotating Disc Extractor.* VERMIJS and KRAMERS(22) have presented data for the extraction of acetic acid from methyl isobutyl ketone with water, using a small rotating disc extractor as shown in Fig. 20.19. Some of their results are shown in Fig 20.20, where $K_D . \alpha$ is plotted versus the ratio of the flow rates of the two phases $V_D/V_C$, the water phase being dispersed. $\alpha$ is here the total area of interface, and $K_D . \alpha$ is found from

$$V_D . C = K_D . \alpha(\Delta C)_{\text{lm}} \qquad \ldots .(20.31)$$

$V_D$ is the volumetric flow of the dispersed phase, and
$C$ is the concentration in the water phase leaving extractor.

From these relatively limited results, it appears that this rotating disc unit has a similar efficiency to a multi-stage mixer, but is probably rather less efficient than the Scheibel column, referred to later.

## EQUIPMENT FOR EXTRACTION

The simplest classification of the various forms of equipment is between those in which the compositions of the phases change continuously, and those in which the change is stagewise. The first group are referred to as differential contact equipment, and the second as stagewise equipment. The former type will now be discussed in more detail.

TABLE 20.2. *Miscellaneous spray-tower extraction data*

| Dispersed phase | Continuous phase | Extracted solute | Direction of extraction | Tower diam., in. | Distributor nozzles | | $u_C$ | $u_D$ | Equation $Ka$ in hr$^{-1}$ **H** in ft |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | No. | *I.D.* in. | Ft/hr | | |
| Water | Benzene | Acetic acid | $D \rightarrow C$ | 1·824 | 36 | 0·10 | 44·1–45·3 | 44–175 | $K_Ca = 0{\cdot}158u_D$<br>$\mathbf{H}_{OC} = 1{\cdot}73$ to $6{\cdot}34$ |
| Benzene | Water | Acetic acid | $C \rightarrow D$ | 1·824 | 36 | 0·10 | 16–45·4 | 44·6–120 | $K_Da = 0{\cdot}157u_D$<br>$\mathbf{H}_{OD} = 5{\cdot}4$ to $10{\cdot}9$ |
| Benzene | Water | Acetic acid | $C \rightarrow D$ | 3·55 | 6 | 0·12 | 10–60 | 30 | $\mathbf{H}_{OD} = 3{\cdot}5\left(\frac{u_D}{u_C}\right)^{0{\cdot}132}$ |
| Water | Methyl isobutyl ketone | Benzoic acid | $C \rightarrow D$ | 1·824 | 36 | 0·10 | 46–46·9 | 43·7–175 | $K_Ca = 0{\cdot}00665u_D$<br>$\mathbf{H}_{OC} = 37{\cdot}5$ to $157$ |
| Water | Methyl isobutyl ketone | Propionic acid | $C \rightarrow D$ | 1·824 | 36 | 0·10 | 43·9–46·0 | 15·9–132 | $K_Ca = 0{\cdot}267u_D$<br>$\mathbf{H}_{OC} = 1{\cdot}18$ to $10{\cdot}37$ |
| Methyl isobutyl ketone | Water | Propionic acid | $C \rightarrow D$ | 1·824 | 36 | 0·10 | 44·2–45·0 | 49·6–132·0 | $K_Da = 0{\cdot}281u_D$<br>$\mathbf{H}_{OD} = 3{\cdot}56$ |
| Methyl ethyl ketone | $CaCl_2$ brine | Water | $D \rightarrow C$ | 3·55 | 6 | 0·12 | 16·1–35·6 | 26·7–66·1 | $K_Da = 0{\cdot}4u_D$<br>$\mathbf{H}_{OD} = 2{\cdot}5$ |

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## Differential Contact Equipment

Some of the equipment in this group relies on gravitational separation of the two phases. It is represented by spray, baffle plate, and packed columns, all of which are relatively cheap in first cost and require little maintenance. On the other hand, they are relatively low in efficiency and are only really suitable where a small number of stages are required. The others incorporate some mechanical agitator or separator.

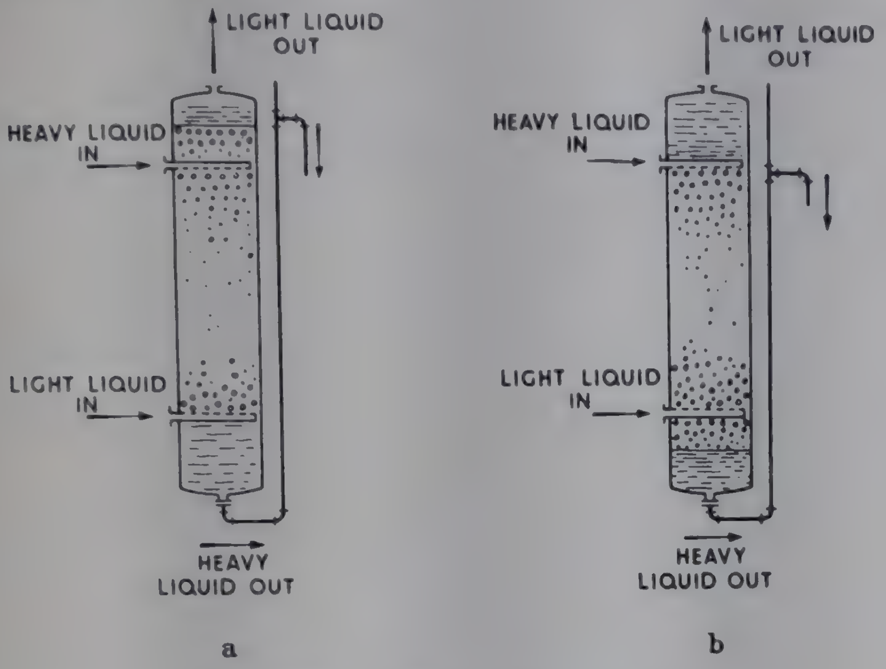

Fig. 20.21. Spray columns (*a*) (*b*)

(1) *Spray Column.* Two methods of operating spray columns are shown in Fig. 20.21*a* and *b*. In the simplest form, the spray enters from the distributor at the bottom and the droplets rise up to the top and then pass out; the heavier liquid flows in the reverse direction down the column. The efficiency is very low, and up to 20 feet may be required to obtain one theoretical stage.

(2) *Baffle-Plate Columns.* These are simple cylindrical columns provided with baffles to direct the flow of the dispersed phase. This is shown in Fig. 20.22. The efficiency of each plate is very low, but since the baffles can be put very close together, 3 in. to 6 in., it is possible to obtain several theoretical stages in a reasonable height.

(3) *Packed Columns.* These are really identical with those used for distillation and absorption, and are filled with Raschig rings or other small packings. The packing reduces the recirculation of the continuous phase, and helps in the redistribution of the dispersed phase. The drawbacks to these columns are that they will not take fluids containing solids in suspension, that they have a relatively low throughput, and that, as the diameter is increased, the efficiency is substantially reduced. Their simplicity of construction and operation have however led to their wide use for low flow rates.

As with gas absorption, in the operation of packed columns the flow rates should be just below those required to cause flooding. DELL and PRATT$^{(16)}$

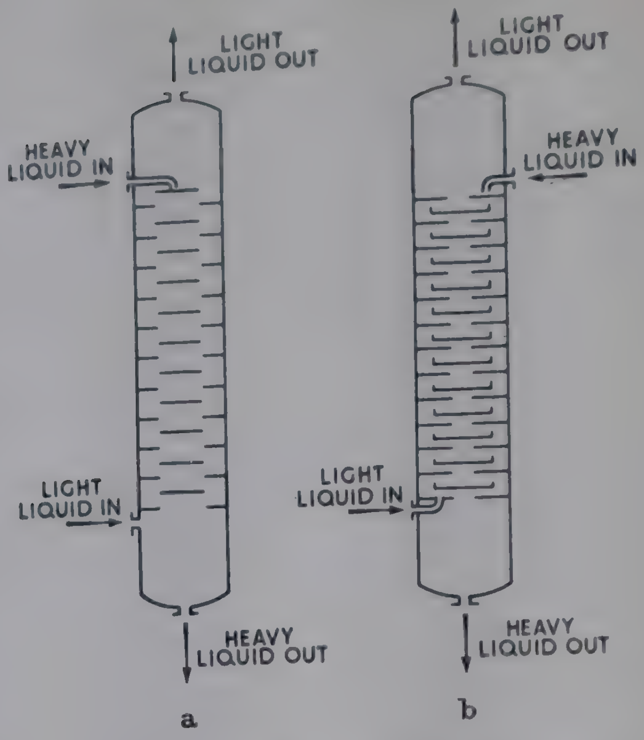
LIGHT LIQUID OUT
HEAVY LIQUID IN
LIGHT LIQUID IN
HEAVY LIQUID OUT
a
LIGHT LIQUID OUT
HEAVY LIQUID IN
LIGHT LIQUID IN
HEAVY LIQUID OUT
b

Fig. 20.22. Baffle-plate column (*a*) (*b*)

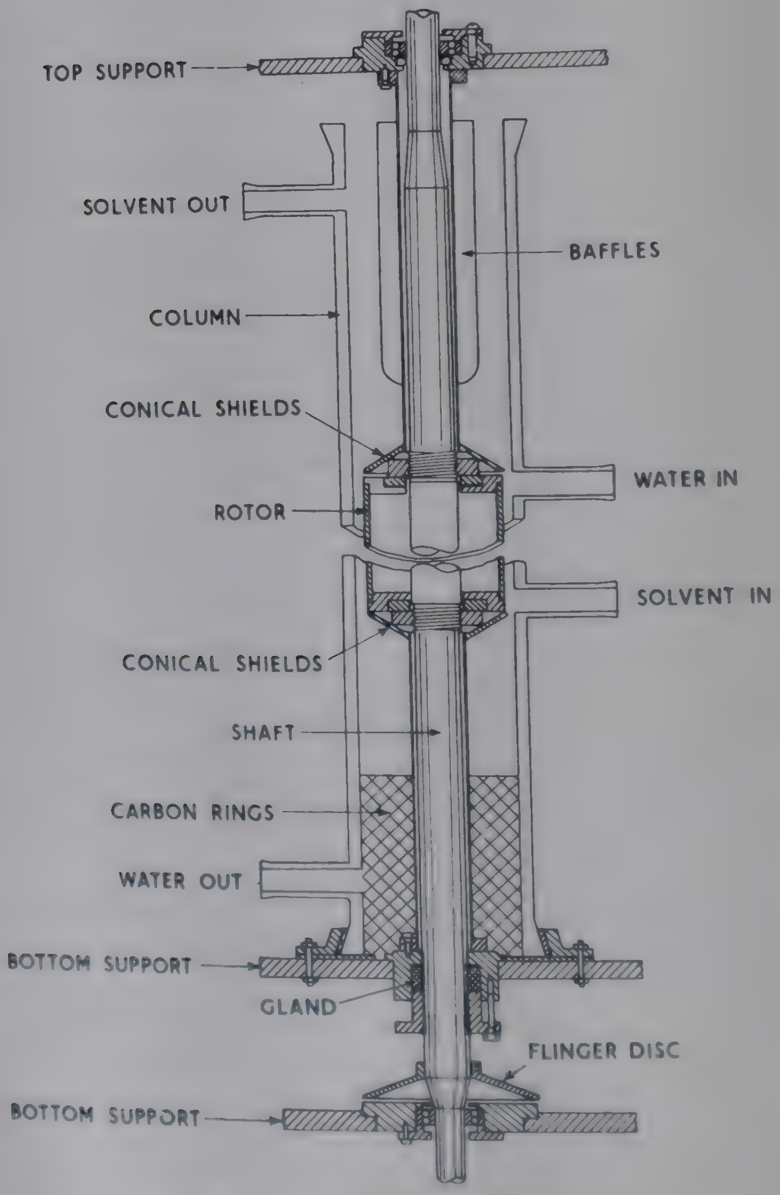
TOP SUPPORT
SOLVENT OUT
BAFFLES
COLUMN
CONICAL SHIELDS
WATER IN
ROTOR
SOLVENT IN
CONICAL SHIELDS
SHAFT
CARBON RINGS
WATER OUT
BOTTOM SUPPORT
GLAND
FLINGER DISC
BOTTOM SUPPORT

Fig. 20.23. Rotary annular column

have examined the flooding rates in columns of 3 to 6 in. diameter for two-phase flow, and have shown that their results can be correlated by equation 11.25 (Chapter 11). It was found by BLANDING and ELGIN(9) that the flooding rates for a given system could be expressed by the relation

$$u_C^{\frac{1}{2}} + u_D^{\frac{1}{2}} = \text{constant} \qquad \ldots .(20.32)$$

where $u_C$ and $u_D$ are the mean linear velocities of the two phases.

(4) *Rotary Annular Columns*(21). With these columns, mechanical energy is provided to break down the dispersed phase. This permits a much finer degree of dispersion to be obtained and results in an improved efficiency. An illustration of this type of column is given in Fig. 20.23. An inner rotating cylinder gives rise to vortex formation between the wall and the rotor, and the shear forces which are set up break down the dispersed phase into very small droplets. This type of unit has not been developed for very large installations. In a modified form of the apparatus, the vortices are set up by the rotation of the discs and are confined to the space between the annular baffles; a very good performance is obtained. The equipment is particularly suitable for installations where a moderate number of stages are required, and where the throughput is considerable.

(5) *Pulsed Column*. In order to prevent coalescence of the dispersed drops, DIJCK(5) and others have devised methods of giving the whole of the continuous phase a pulsed motion. This can be done either by some mechanical device, or by the introduction of compressed air.

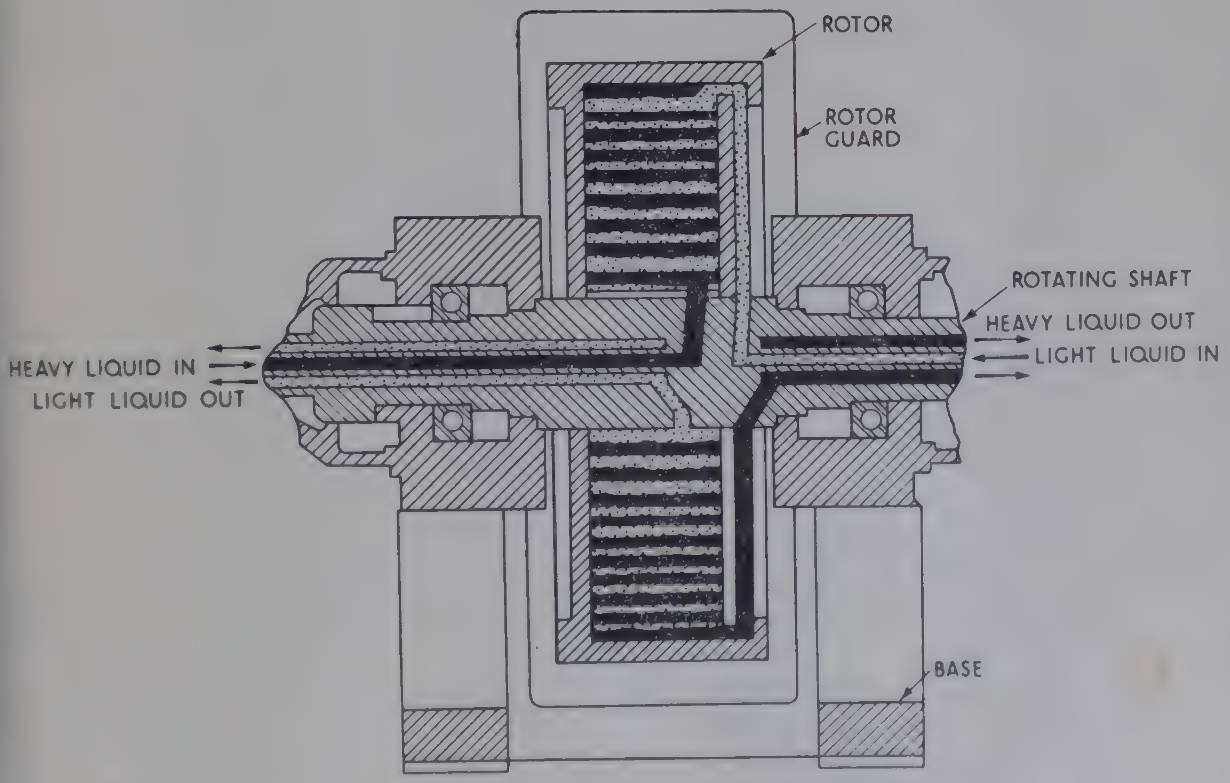

Fig. 20.24. Podbielniak extractor

(6) *Podbielniak Extractor.* This is a continuous extractor using centrifugal forces to separate the two phases. A rotor, in the form of a long perforated ribbon, is coiled into a spiral and is rotated at high speed inside a fixed casing. A long passage is provided for the flow of the two phases, as indicated in Fig. 20.24. This type of unit has a high capacity for its size and a low hold-up. It will handle systems with a very small density difference between the phases, and is suitable where emulsification tends to occur. The high first cost and the problem of maintenance are drawbacks, but the equipment has been found useful for the handling of antibiotics.

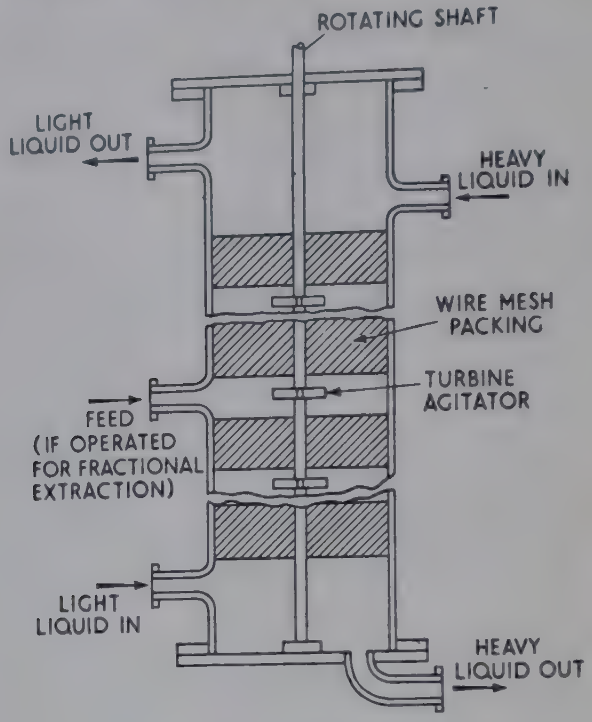
ROTATING SHAFT
LIGHT LIQUID OUT
HEAVY LIQUID IN
WIRE MESH PACKING
TURBINE AGITATOR
FEED (IF OPERATED FOR FRACTIONAL EXTRACTION)
LIGHT LIQUID IN
HEAVY LIQUID OUT

Fig. 20.25. Scheibel Column

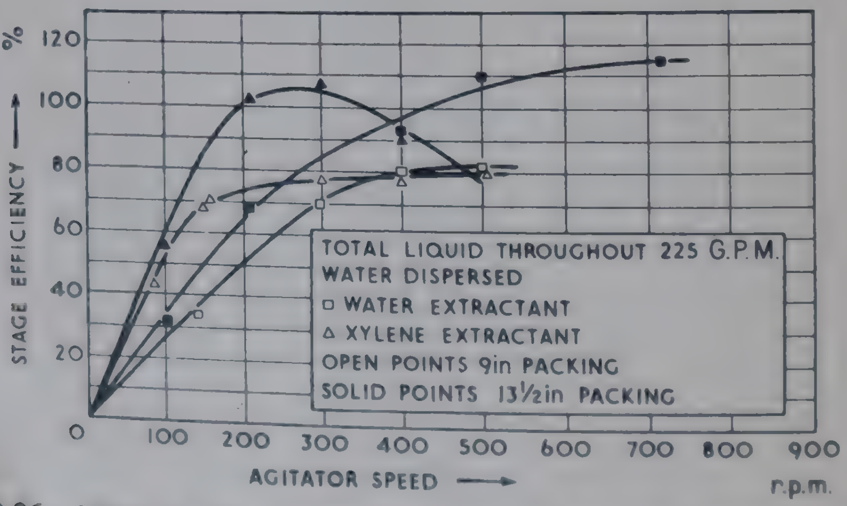

Fig. 20.26. Effect of agitator speed on efficiency. System acetone-xylene-water

(7) *The Scheibel Column.* One of the difficulties in using perforated plate or packed columns is that redispersion of the liquids after each stage is very poor. To overcome this, SCHEIBEL and KARR[13] introduced a unit, as indicated in

Fig. 20.25, in which a series of agitators is mounted on a central rotating shaft. Between the agitators is fitted a wire mesh section which successfully breaks up any emulsions. Some results for a column 11½ in. diameter, with 4 in. diameter agitators and with packing sections of 9 and 13½ in., are shown in Fig. 20.26. Results are given in the paper for the two systems, water-xylene-acetone, and water-xylene-acetic acid. In both cases, it is found that one theoretical stage is obtained in a height of between 1·5 to 2·5 ft; this is a big improvement on that usually obtained in a packed column. Although there are few data on the use of large units, there should be no falling off in efficiency as the diameter is increased.

## MECHANISM OF MASS TRANSFER

In the previous chapter, some account was given of two possible mechanisms for mass transfer; namely, the two-film theory of Whitman(3), and the penetration theories of Higbie(4) and Danckwerts(15). The same kind of problem occurs in liquid-liquid extraction where we are dealing with a dispersed liquid phase and a continuous liquid phase. The passage of the dispersed drops through the unit requires first of all their formation at a nozzle, and secondly their collapse at some boundary such as a perforated plate, at the other side of which the drops reform. The general equations given for the calculation of the rate of extraction in a column do not take into consideration these three stages of the process. The process may occur only once, as in short spray towers, or it may be repeated several times, as in perforated plate or packed towers. Sherwood(7) passed drops of benzene or methyl isobutyl ketone containing a small percentage of acetic acid into a column of water, and measured the concentrations of the inlet and outlet streams. By plotting the ratio of the unextracted solute to the total solute which could be extracted if equilibrium were attained, against the height of the column, he found that up to 40% of the extraction occurred during the formation of the drops. Licht and Conway(14) allowed drops of water containing acetic acid to fall through a small column containing isopropyl ether, methyl isobutyl ketone, or ethyl acetate. By plotting the percentage of acid extracted against height, as in Fig. 20.27, and extrapolating the curve, it appeared that up to 30% of the extraction occurred during the formation of the drops.

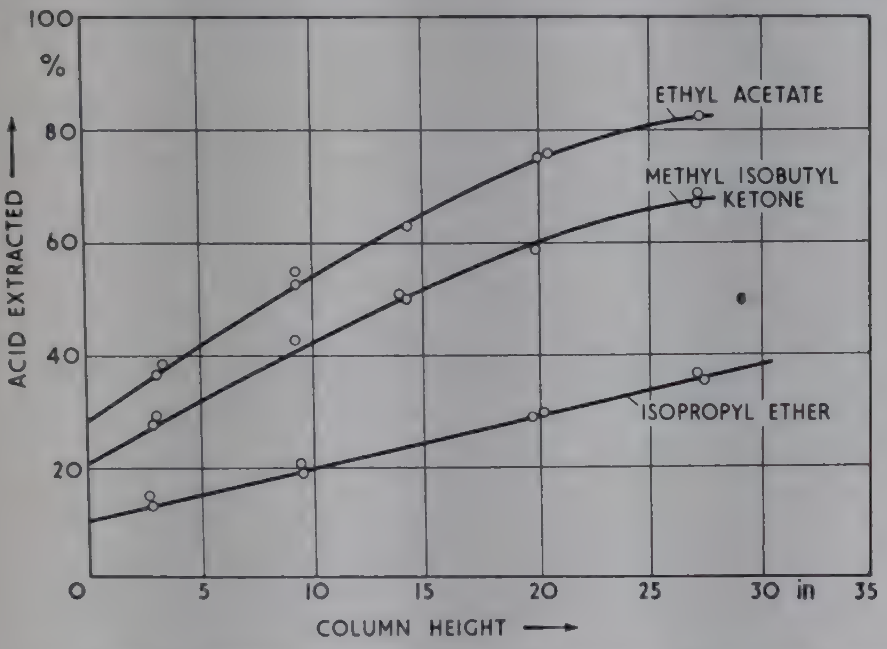

Fig. 20.27. Percentage extraction as a function of height of column
(Extraction of acetic acid from water with various solvents.)

Coulson and Skinner(18) have measured the rate of transfer to a drop during its rise in a still liquid, and to drops rising against a falling continuous phase. They worked with drops of 0·5 to 0·1 ml. formed in a vertical glass tube of $1\frac{1}{2}$ in. diameter. Some of the results for drops of benzene formed in 0·5N propionic acid are shown in Fig. 20.28, where the percentage approach to equilibrium attained by the drop is plotted against the time of formation and collapse of the drop ($t_f$). It is seen that equilibrium is more nearly approached as the time of formation and collapse are increased, but that for $t_f$ greater than 4 seconds, the increase is very small. It was found that the transfer coefficient varied approximately as $t_f^{-0\cdot7}$. The transfer was greater than would be expected, on the assumption that the drop rises as a solid sphere, but was less than that which would be obtained with a rapid replacement of the material at the interface. Thus, if we consider a point $A$ on the surface of the drop, a point $B$ in the surrounding medium and $C$, the centre of the drop, three possibilities occur in the relative position of $A$, $B$, and $C$ as the drop rises to some point shown in Fig. 20.29.

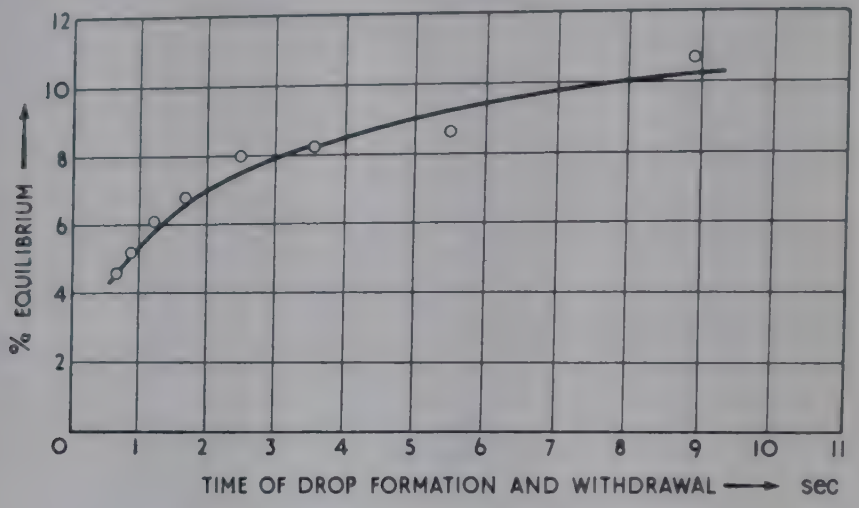

Fig. 20.28. Approach to equilibrium as a function of time of formation and collapse of drop

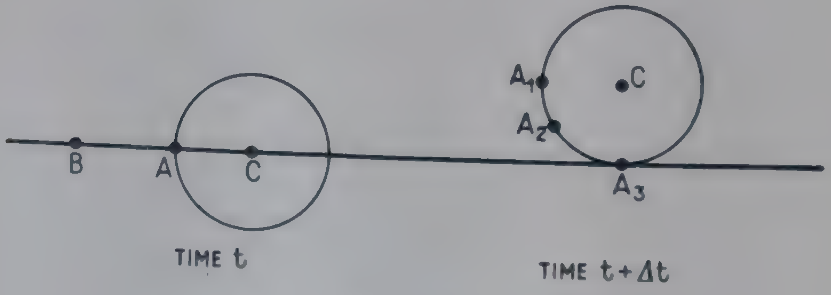

Fig. 20.29. Drop rising in liquid

$A_1$ Here points $A$ and $C$ have remained in the same relative position, the drop rising as a solid sphere.

$A_3$ $A$ and $B$ remain in the same relative positions, $A$ moving downwards relative to $C$ at a velocity equal to the rate of rise of the drop. Thus the interface will be replaced each time the drop moves through its own height.

$A_2$ $A_2$ is a point somewhere between $A_1$ and $A_3$.

In one example using propionic acid, they were able to show that the actual approach to equilibrium was 55%, whereas on the assumption of the drop acting as a solid sphere, it would only be 19%.

The movement inside drops has been examined by GARNER and HALE$^{(19)}$, and it is fairly clear that some form of internal recirculation takes place.

In liquid-liquid extraction, the transfer is brought about by the active movement of a dispersed drop in a continuous phase, and studies with individual drops do provide the best information on the actual mechanism of the process. In perforated plate columns the drops break up and then reform fairly frequently, whereas in packed columns the drop may stay roughly in its initial form for a considerable period.

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## LIST OF SYMBOLS USED IN CHAPTER 20

| | | |
|---|---|---|
| $A$ | Mass of solvent $A$ | M |
| $a$ | Interfacial area per unit volume | $L^{-1}$ |
| $B$ | Mass of solvent $B$ | M |
| $C$ | Concentration of solute | $ML^{-3}$ |
| $C^*$ | Value of concentration in equilibrium with second phase | $ML^{-3}$ |
| $E$ | Mass of extract | M |
| $F$ | Mass of feed | M |
| $\mathbf{H}$ | Height of the transfer unit | L |
| $K$ | Overall mass transfer coefficient | $LT^{-1}$ |
| $K'$ | Distribution constant | — |
| $k$ | Mass transfer coefficient | $LT^{-1}$ |
| $L$ | Volumetric rate of flow per unit area | $LT^{-1}$ |
| $M$ | Total flow of material through the system | M |
| $m$ | Slope of the equilibrium line | — |
| $N$ | Molar rate of transfer per unit area | $ML^{-2}T^{-1}$ |
| $\mathbf{N}$ | Number of transfer units | — |
| $n$ | Number of stages | — |
| $P$ | Difference stream | M |
| $R$ | Mass of raffinate | M |
| $S$ | Added solvent in extract stream | M |
| $s$ | An index | — |
| $t_f$ | Time of formation and collapse of drop | T |
| $u$ | Volumetric flow rate per unit area | $LT^{-1}$ |
| $V$ | Volumetric flow rate | $L^3T^{-1}$ |
| $X_f$ | Mass ratio of solute in feed | — |
| $X_1$, $X_2$ | Mass ratio of solute in raffinate | — |
| $x_A$, $x_B$ | Mass or mol fraction of $A$, $B$ | — |
| $Y_1$, $Y_2$ | Mass ratio of solute in extract | — |
| $Z$ | Height of packing | L |
| $\alpha$ | Total interfacial area | $L^2$ |
| $\beta$ | Selectivity ratio | — |
| $\phi$ | A constant | — |

Suffixes

| | |
|---|---|
| $C$, $D$ | continuous and disperse phases |
| $E$, $R$ | extract and raffinate phases |
| $O$ | overall (transfer units) |
| $i$ | value at interface |
| lm | logarithmic mean value |
| 1, 2 | values at bottom, top of column |

## Section H

# *Evaporation Crystallisation and Drying*

## CHAPTER 21

# *Evaporation*

EVAPORATION is one of the main methods used in the chemical industry for the concentration of aqueous solutions. The usual meaning of the term is the removal of water from a solution by boiling the liquor in a suitable vessel, the evaporator, and withdrawing the vapour. If the solution contains dissolved solids, the resulting strong liquor may become saturated so that crystals are deposited.

The various liquors to be evaporated can be classified in the following ways:

(1) Those that can be heated to high temperatures without decomposition, and those that can only be heated to a low temperature (130°F or 55°C).

(2) Whether or not they give rise to solids on concentration; here crystal size and shape may be important.

(3) Those which at any given pressure boil at about the same temperature as water, and those which have a much higher boiling point.

Evaporation is carried out by adding heat to the solution to vaporise the solvent. The heat is supplied largely to provide the latent heat of vaporisation, and, by adopting methods for recovery of heat from the vapour, the chemical engineer has been able to bring about great economy in heat utilisation. Whilst the normal heating medium is generally low pressure steam, for special purposes Dowtherm or flue gases may be adopted.

The design of an evaporation unit requires the practical application of our ideas of heat transfer to boiling liquids, together with a realisation of what happens to the liquid during concentration. In addition to the three main features outlined above, we must be particularly careful with liquors which have an inverse solubility curve and which are therefore likely to deposit scale on the heating surface.

The type of equipment used depends largely on the method of applying heat to the liquor and the method of agitation. Heating may be either direct or indirect. Direct heating is represented by solar evaporation and by submerged combustion of a fuel. In indirect heating, the heat, generally provided by the condensation of steam, passes through the heating surface of the evaporator.

## EVAPORATORS WITH DIRECT HEATING

The use of solar heat for the production of Glauber's salt at Lake Chaplin has recently been described by HOLLAND(8, 11). The brine is pumped in hot weather to reservoirs of a million square feet area to a depth of ten to fifteen feet, and salt is deposited. Later in the year, the mother liquor is drained off and the salt stacked mechanically, and conveyed to special Holland evaporators. These are large vessels (10 × 12 × 14 ft); hot gases enter at 1600–1800°F (900–1000°C) through a suitable refractory duct and leave at about 140°F (60°C). The salt crystals melt in their own water of crystallisation and are then dried in the stream of hot gas. Bloch and associates(13) have examined the mechanism of evaporation of salt brines by direct solar energy. They found that the rate of evaporation increased with the depth of brine; thus, a 50-cm depth of brine gave a 16% higher rate than a 20-cm depth. The addition of dyes, such as 2-naphthol green, enables the solar energy to be absorbed in a much shallower depth of brine, and this technique has been used to obtain a marked increase in the rate of production in the Dead Sea area.

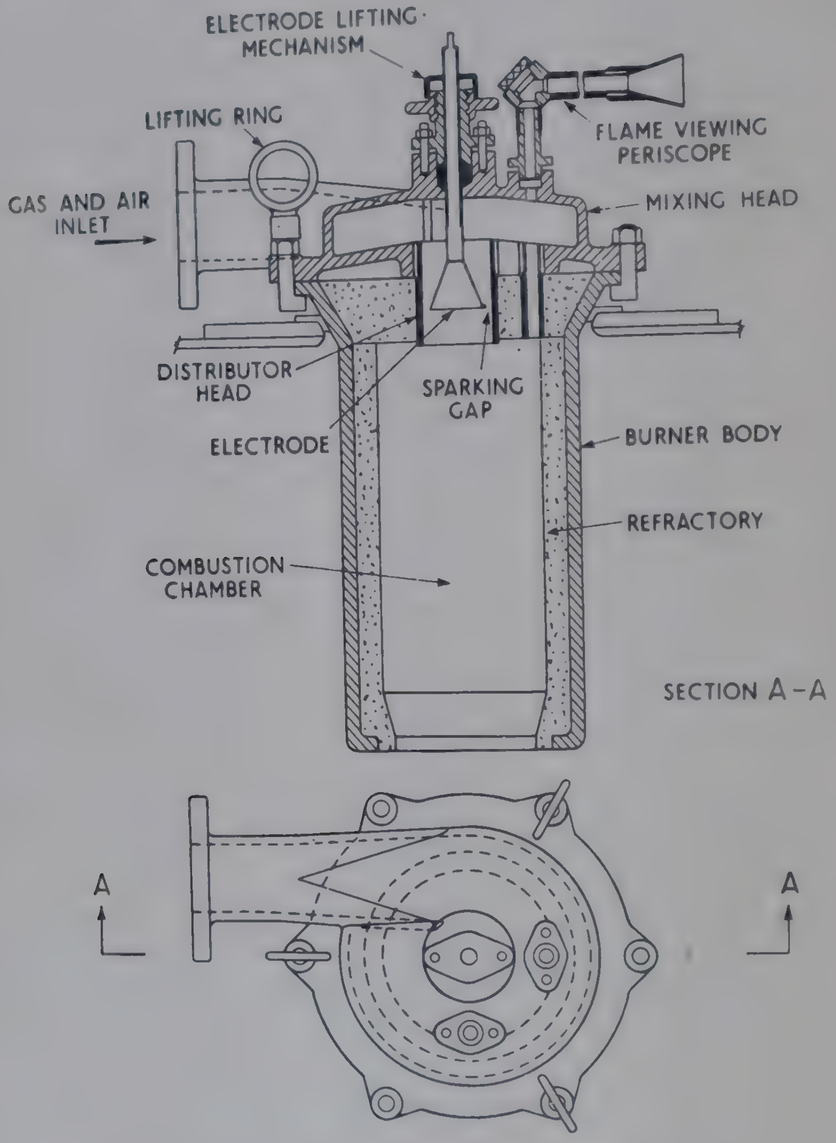
ELECTRODE LIFTING MECHANISM
LIFTING RING
FLAME VIEWING PERISCOPE
GAS AND AIR INLET
MIXING HEAD
DISTRIBUTOR HEAD
SPARKING GAP
ELECTRODE
BURNER BODY
REFRACTORY
COMBUSTION CHAMBER
SECTION A-A
A
A

Fig. 21.1. Burner for submerged combustion

The submerged combustion of a gas, such as coal gas, has been used for the concentration of very corrosive liquors, including spent pickle liquors, weak phosphoric and sulphuric acids. A suitable burner for direct immersion in the liquor, as developed by Swindin(9), is shown in Fig. 21.1. The depth of immersion of the burner is determined by the time of heat absorption; thus, a

2-in. burner may be immersed 10 in. and a 7-in. burner about 18 in. The efficiency of heat absorption is measured by the difference between the temperature of the liquid and that of the gases leaving the surface, values of 2 to 5°C being obtained in practice. The great attraction of this technique, apart from the ability to handle corrosive liquors, is the very great heat release obtained and the almost instantaneous transmission of the heat to the liquid. Thus SWINDIN quotes 7,000,000 B.Th.U/ft$^3$-hr (4,000,000 lb-cal/ft$^3$-hr) as a practical figure.

## STEAM-HEATED EVAPORATORS

Whilst each of the above methods is of considerable importance in a given industry, it is the steam-heated evaporator that is the main type of unit in the chemical industry; this will be considered in detail. We are here concerned with the transfer of the latent heat from steam to a boiling liquid. In Chapter 6, on heat transfer, it was shown that the movement of the liquid over the heating surface had a marked influence on the rate of heat transfer, and it is thus convenient to classify evaporators according to the method of agitation or the nature of the circulation of the liquor over the heating surface. On this basis they may be divided into three main types:

(1) Natural circulation units.

(2) Forced circulation units.

(3) Film-type units.

The developments that have taken place in design have, in the main, originated from the sugar and salt industries where the cost of evaporation represents a major item in the process. In recent years, particular attention has been given to obtaining the most efficient use of the heating medium, and the main techniques that have been developed are the use of the multiple effect unit, and of various forms of vapour compression units.

### Natural Circulation Units

With these evaporators, circulation of the liquor is brought about by convection currents arising from the heating surface. This group may be sub-divided according to whether the tubes are horizontal with the steam inside, or vertical with the steam outside.

***Horizontal Tubes.*** N. RILLIEUX is usually credited with the idea of using horizontal tubes, and a modern unit of this type is as shown in Fig. 21.2. The horizontal tubes extend between two tube plates to which they are fastened either by packing plates or by expansion. One method, as used by the Swenson Evaporator Company, is indicated in Fig. 21.3; the holes in the tube sheets are countersunk to receive conical gaskets which are fitted over the ends of the tubes, and are forced in by the packing plates mounted on studs from the tube plate. This form of construction enables quick replacement of the tubes to be carried out, but in many installations expanding of the tubes is satisfactory. Above the heating section is a cylindrical portion in which separation of the vapour from the liquid takes place. The vapour leaves through some form of de-entraining device to prevent the carry-over of liquid droplets with the vapour stream. The steam enters one steam chest, passes through the tubes and out into the opposite chest, and the condensate leaves through some form of steam trap.

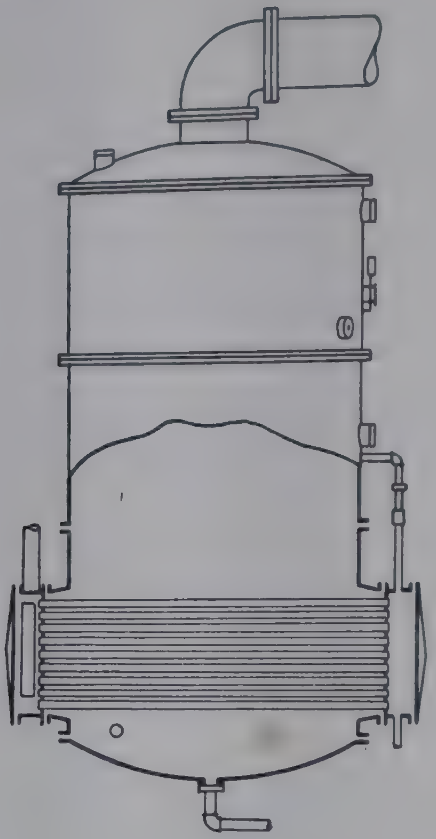

Fig. 21.2. Natural circulation evaporator with horizontal tubes

These horizontal evaporators are relatively cheap, require low head room, and are easy to install. They are suitable for handling liquors that do not crystallise, and give good heat transfer. They can be used either as batch or as continuous units. The body is generally from 3 to 12 ft diameter and 8 to 13 ft high, and the tubes generally range from $\frac{7}{8}$ to $1\frac{1}{4}$ in. diameter. The liquor circulation is poor, and for this reason they are unsuitable for viscous liquors.

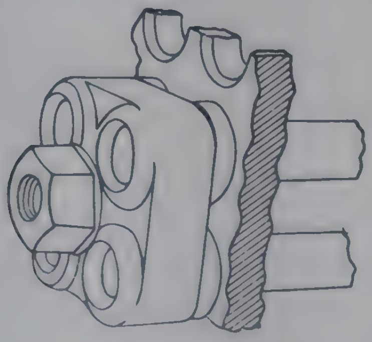

Fig. 21.3. Method of securing tubes to tube plate in Swenson evaporator

***Vertical Tubes.*** The use of vertical tubes is associated with Robert, a director of a sugar factory in Austria, so that this type is sometimes known as the Robert or Standard Evaporator. A modern form of vertical evaporator is illustrated in Fig. 21.4, in which it will be seen that a vertical cylindrical body is used, with the tubes held between two horizontal tube plates which extend right across the body; the lower portion of the evaporator is frequently spoken of as the calandria section (Fig. 21.9). The early plants of this type consisted of a very large number of tubes covering the whole cross-section of the body. It was found, however, that the circulation was poor for, although it was easy to generate upward currents, it was difficult for the liquor to return to the bottom.

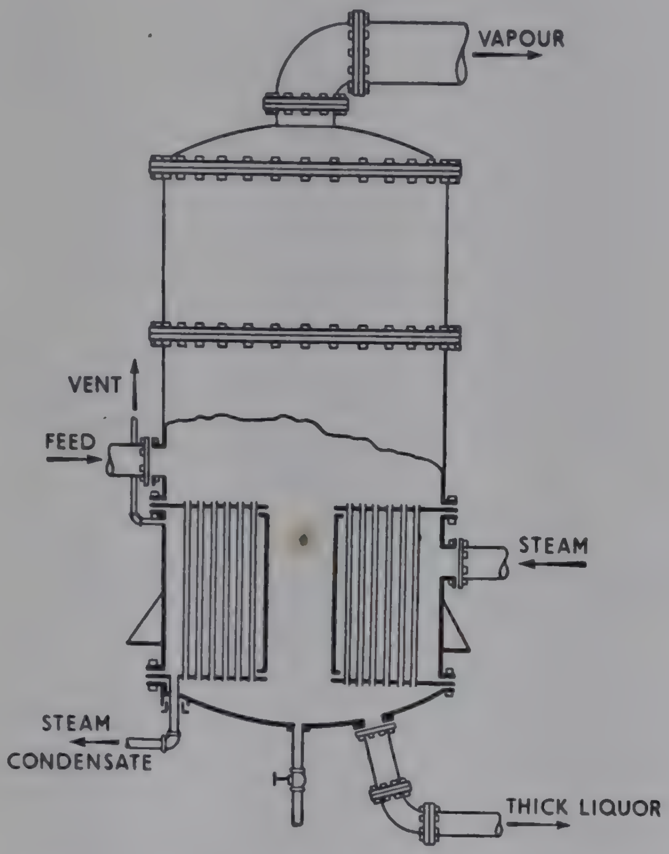

Fig. 21.4. Evaporator with vertical tubes and large central downcomer

This layout was improved by Kasalovsky who introduced the large central downtake tube which enables good circulation to be obtained. In this form of evaporator, the liquor level is usually maintained just above the top of the tubes. The area of this central tube varies from about 40% to 100% of the total cross-sectional area of the tubes. The Scott Company have used several tubes, of wider diameter than the main tubes, to serve as a number of downcomers. Again, other firms employ downcomers at the side of the body. The tubes range from 3 to 6 ft in length and from 1½ to 3 in. diameter, giving a ratio of length to inside diameter of the tubes of between 20 and 40. In 1877, the basket type (Fig. 21.5) was introduced; in this, vertical tubes are still used with the steam outside, but the heating element is suspended in the body so as to give an annular downtake. The advantages claimed for this construction are that the heating unit is easily removed for repairs, and that crystals formed in the downcomer do not break up. As the circulation of the liquor in the tubes is better, the vertical tube evaporator is the standard type. It is widely used in the sugar, salt and heavy chemical industries (i.e. ammonium nitrate, caustic soda), where the throughputs are very large.

***Coil Evaporators.*** For the final concentration of sugars, a coil heating unit has been found very suitable, and a modern form of this construction, as

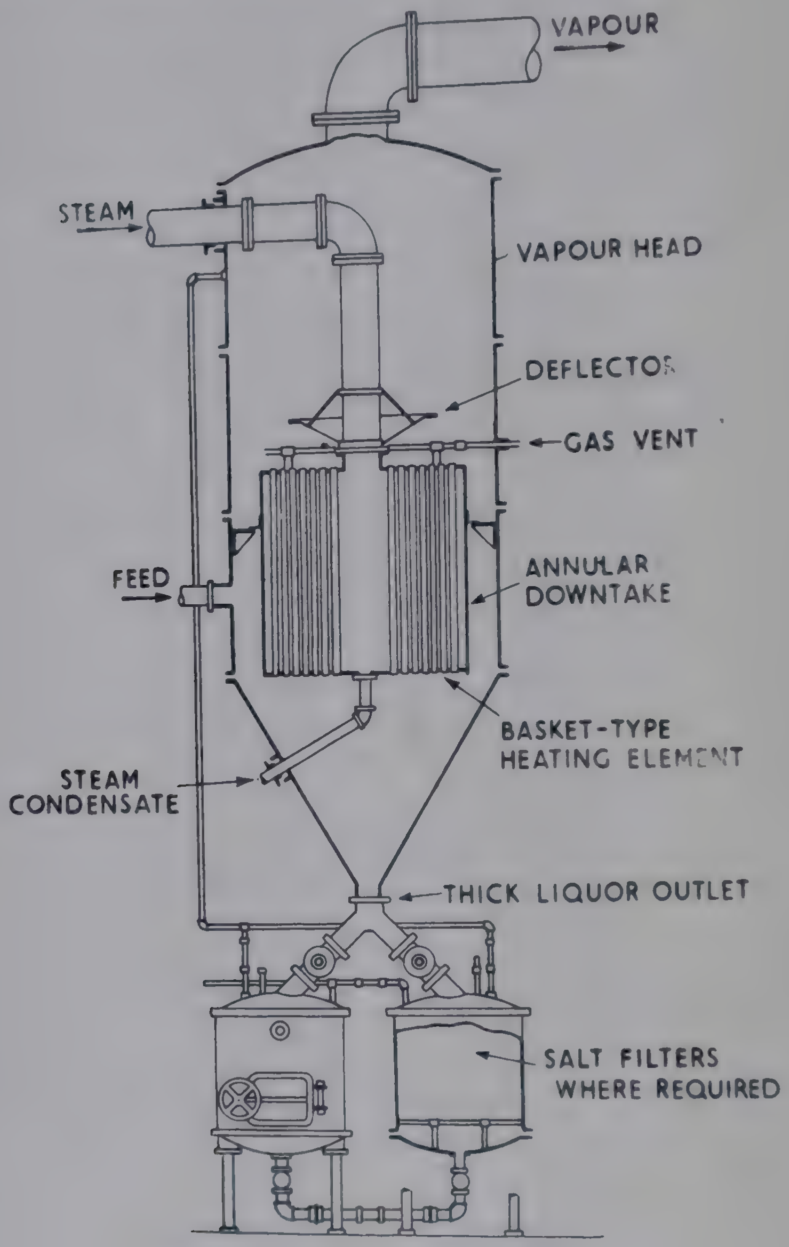
VAPOUR
STEAM
VAPOUR HEAD
DEFLECTOR
GAS VENT
FEED
ANNULAR DOWNTAKE
BASKET-TYPE HEATING ELEMENT
STEAM CONDENSATE
THICK LIQUOR OUTLET
SALT FILTERS WHERE REQUIRED

Fig. 21.5. Basket type evaporator

described by Venton[(15)] is shown in Fig. 21.6. This pan has a capacity of 8500 gallons and a heating surface of 1·44 sq ft per cubic foot, which is quite a high figure. The coils are of copper tubing of 5 in. outside diameter, but are flattened to give an oval shape as indicated. The spacing between the coils should be at least 3 in., and the central well should be from 0·3 to 0·4 of the diameter of the pan. As an alternative, a vertical calandria may be used, as described by Sheardon *et al.*[(16)]. This unit has tubes of 4·0 in. outside diameter, and 4·25 ft high, set in a steam space of 10 ft diameter at the bottom of the pan, and the disengaging space is of 12 ft diameter. This type has a short overall height and a dished bottom; it is sometimes preferred to the coil type because of easier maintenance. Bosworth and Duloy(14) have measured the rate of circulation of the liquor and found figures of 10 cm/sec for the weak sugars.

### The Forced Circulation Evaporator

Increasing the velocity of flow of the liquor through the tubes will bring about a marked increase in the liquid film transfer coefficient. This is achieved in the forced circulation units where a propeller or other impeller is mounted in the central downcomer, or a circulating pump is mounted outside the evaporator body. Thus, in the concentration of strong brines, an internal impeller is often fitted in the downtake, and this form of construction is particularly useful where crystallisation takes place. A turbine impeller, as shown in Fig. 21.10, is sometimes used for this purpose. Forced circulation enables higher degrees of concentration to be achieved, since the heat transfer rate can be kept up in spite of the increased viscosity of the liquid. It also enables scale forming liquors to be handled, since the scale may be cleared from the heating surface by the motion of the liquid. Because the pumping costs increase roughly as the cube of the velocity, the added cost of operation of this type of unit may make it uneconomic; but many forced circulation evaporators are running with a liquor flow through the tubes of from 7 to 15 ft/sec which is a marked increase on the value for natural circulation. Where stainless steel or expensive

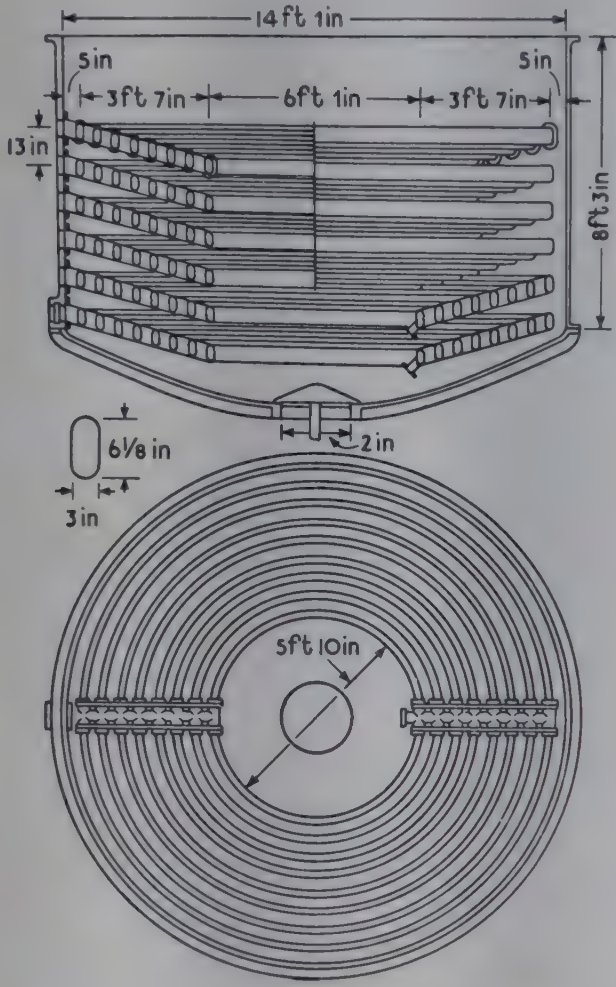

Fig. 21.6. Coil pan of 8500 gal. capacity

alloys such as monel are to be used, forced circulation is favoured because the units can be made smaller and cheaper than those relying on natural circulation. In the type illustrated in Fig. 21.7, there is an external circulating pump; this is usually of the centrifugal type when crystals are present, but vane types may be used in the absence of crystals. The liquor is either introduced at the bottom and pumped straight through the calandria, or else it is introduced in the separating section. In most units, boiling does not take place in the tubes, because the hydrostatic head of liquid raises the boiling point above that in the separating space (see p. 787). Thus the liquor enters the bottom of the tubes and is heated as it rises and at the same time the pressure falls. In the separator the pressure is sufficiently low for boiling to occur. Forced circulation evaporators will work well on such diverse materials as meat extracts, salt, caustic soda, alum or other crystallising material, glues, alcohols and foam-forming materials. A unit with horizontal tubes with two liquid passes through the tubes is shown in Fig. 21.8.

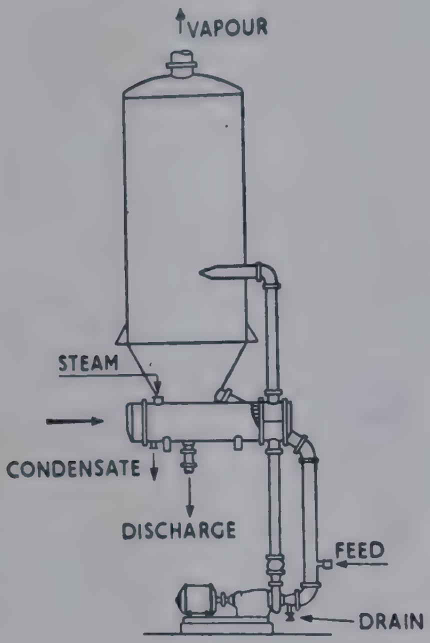

Fig. 21.7. Forced circulation evaporator with external pump

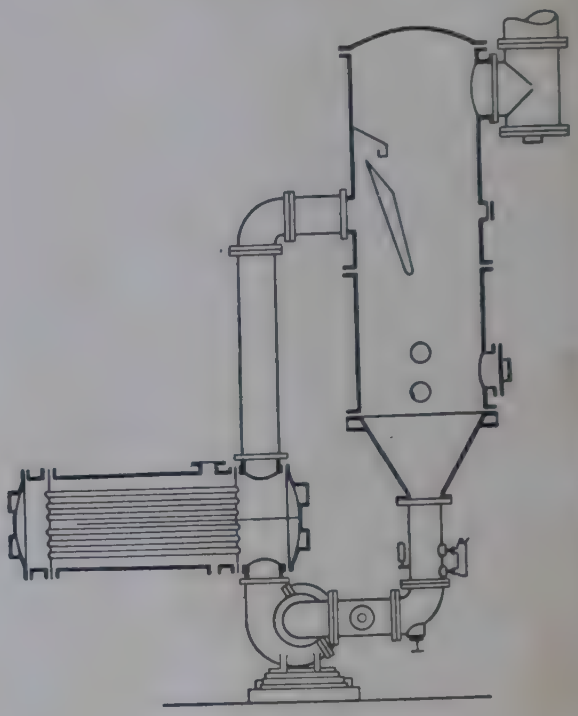

Fig. 21.8. Forced circulation evaporator with horizontal tubes and two liquid passes through the tubes

## Film-Type Units

In all the units so far discussed, the liquor remains some considerable time in the evaporator: with batch operation several hours, and with continuous operation perhaps an hour. This may be undesirable, as many liquors decompose if kept at temperatures at or near their boiling points for any length of time. The temperature can be reduced by operating under a vacuum, as discussed later, but even so there are many liquors which are very heat-sensitive: for example, orange juice, blood plasma, liver extracts and vitamins. If a unit is so constructed that the residence time is only a few seconds, then these

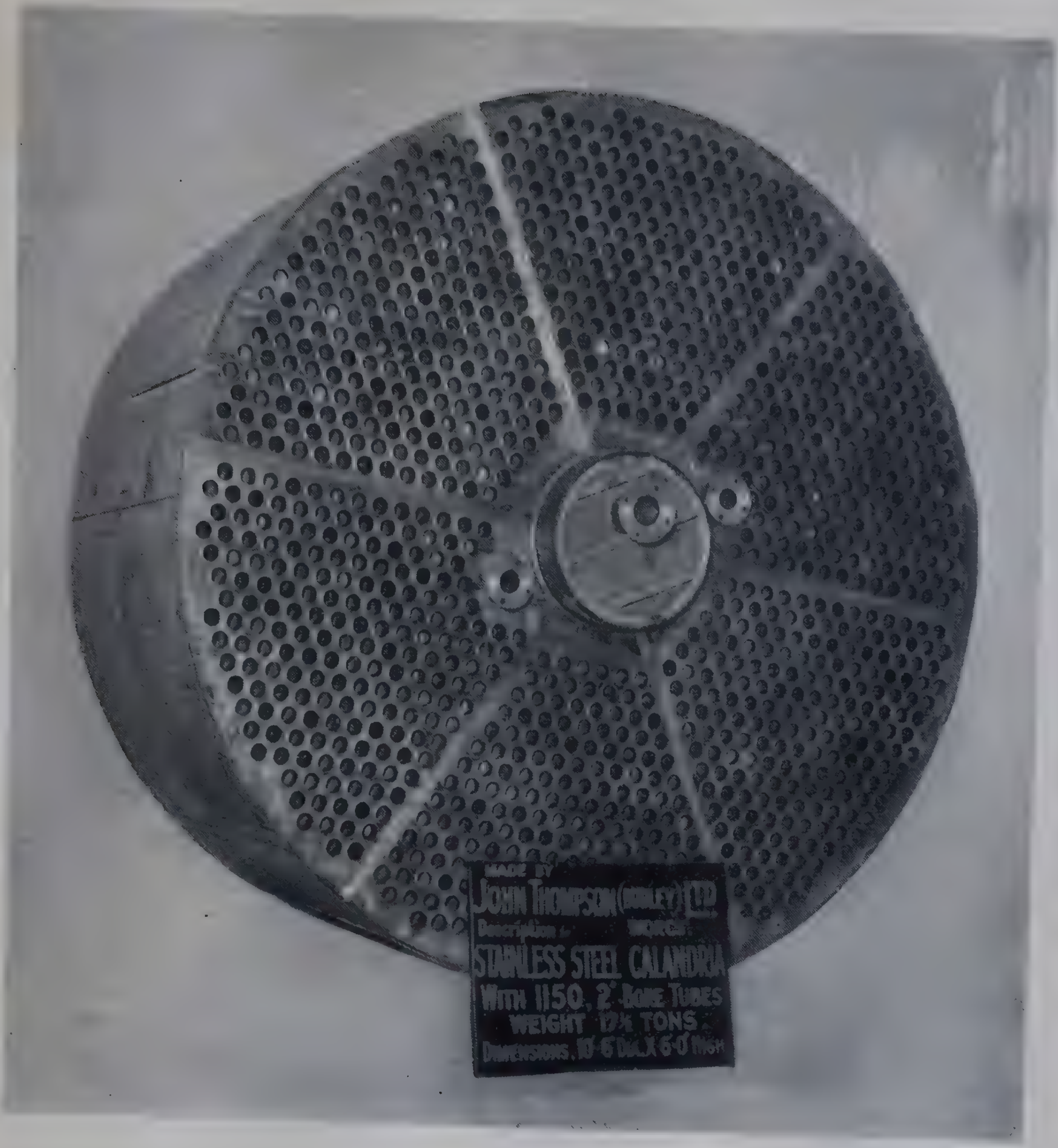
MADE BY
JOHN THOMPSON (DUDLEY) LTD
Description
STAINLESS STEEL CALANDRIA
WITH 1150, 2" BORE TUBES
WEIGHT 17½ TONS
DIMENSIONS, 10'-6" DIA. X 6'-0" HIGH

Fig. 21.9. Calandria of evaporator

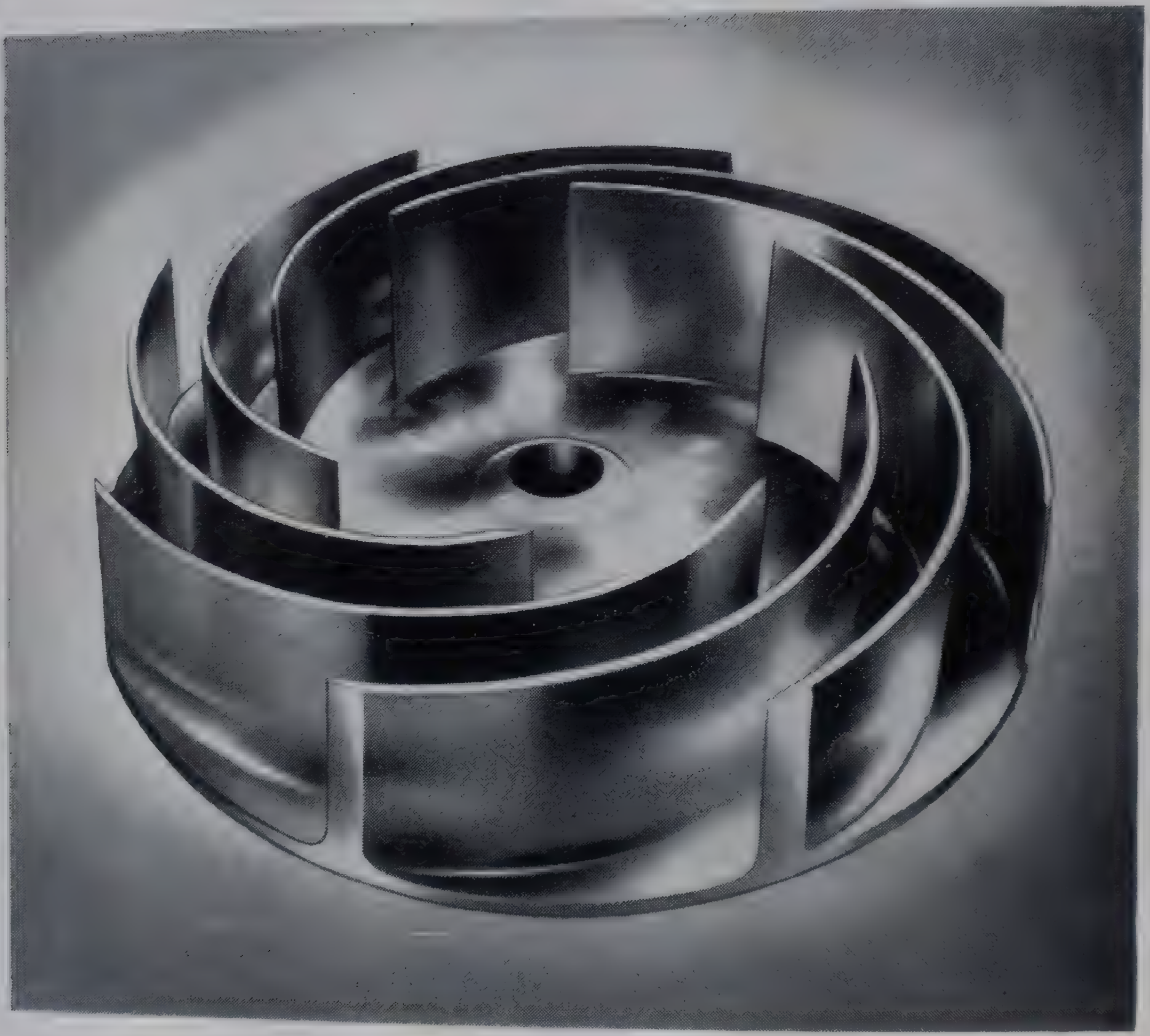

Fig. 21.10. Turbine agitator

dangers are very much reduced. This is the idea behind the Kestner long tube evaporator, introduced in 1909, which is fitted with tubes of $1\frac{1}{2}$ to 2 in. diameter, mounted in a simple vertical steam chest. The liquor enters at the bottom (Fig. 21.11), and a mixture of vapour and entrained liquor leaves at the top and enters a separator, usually of the tangential type. The vapour passes out from the top and the liquid from the bottom of the separator. In the early models the thick liquid was recirculated through the unit, but the once-through system is now normally used. In order to understand the functioning of this type, it is convenient to consider what happens when steam condenses outside a single vertical tube fed at a low rate with cold water. If there is only a small temperature difference between the steam and the water, no boiling will occur and the apparatus will act as a vertical concentric-tube heater. If the temperature difference $\Delta T$ is increased, some bubbles of steam will form near the top, to

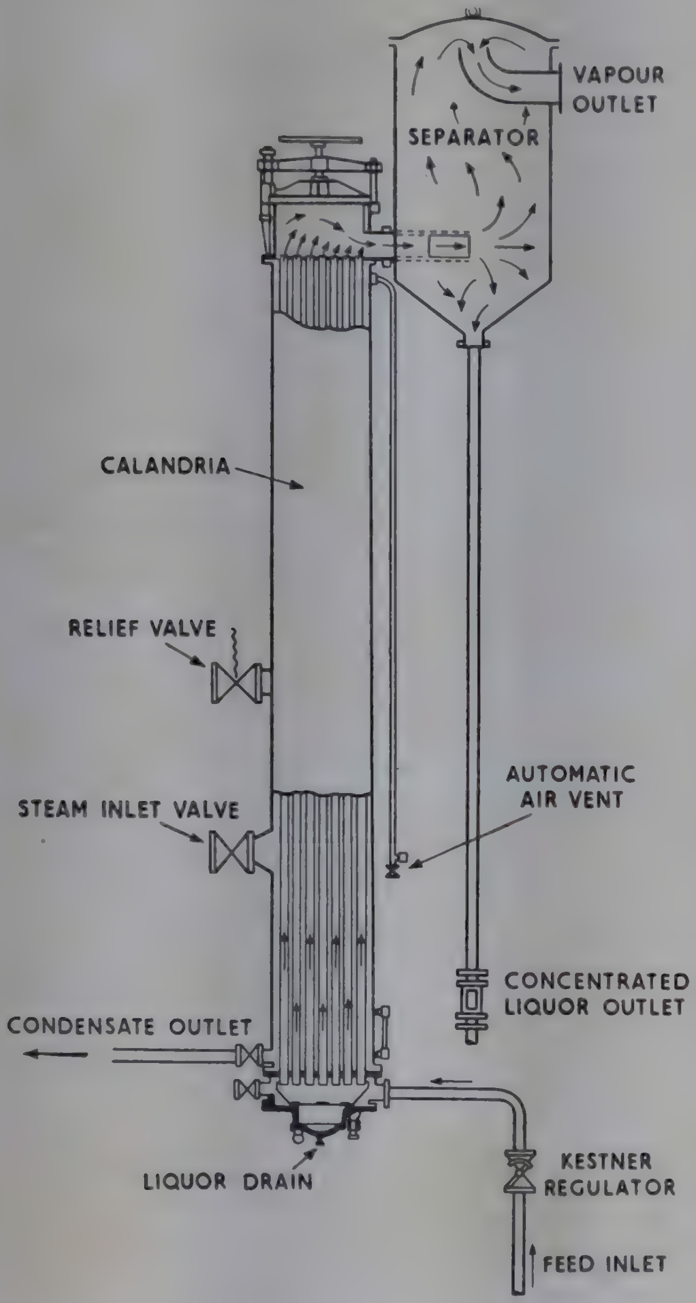
VAPOUR OUTLET
SEPARATOR
CALANDRIA
RELIEF VALVE
AUTOMATIC AIR VENT
STEAM INLET VALVE
CONCENTRATED LIQUOR OUTLET
CONDENSATE OUTLET
LIQUOR DRAIN
KESTNER REGULATOR
FEED INLET

Fig. 21.11. Kestner long tube evaporator

give the appearance of simmering. When $\Delta T$ is further increased, boiling does not occur as with the submerged tubes, but a considerable amount of non-boiling liquid is blown out, followed by a puff of steam. Further increase of $\Delta T$ causes these puffs of liquor and steam to become more frequent and the proportion of steam to rise. Finally, with further increase of $\Delta T$, the slugs are no longer carried up the tube as such, but a column of steam appears to form in the centre of the tube as a result of the expansion of the bubbles, and a film of liquid forms at the walls and is dragged upwards by the high velocity vapour. Drops of water become entrained. A crown of slow-moving drops of liquid forms around the top of the tube. If the temperature difference is sufficiently large, this crown of drops of liquid disappears and, issuing from the top of the tube, is steam containing a suspension of fine particles travelling at very high speed. With such an evaporator, the first portion of the tube will act as a heater, and boiling will not occur until the liquid has risen some distance from the bottom. This is an inefficient arrangement, since the liquid rate will be very low and the rate of heating per unit length of tube correspondingly low. If the heating section is excessively long, then the ratio of height to diameter of the upper section in which boiling occurs will be much too small for satisfactory operation. It is therefore desirable that the feed to this type of unit should be very nearly at its boiling point. The drop in pressure over the tube length will be attributable to the hydrostatic head of the column of non-boiling liquor, together with the friction loss of the steam and water rising in that section where boiling occurs. The first published analysis of the operation of this type of unit was given by BADGER and his associates(5, 6, 7) who arranged for a small thermocouple to be fitted inside the experimental tube ($1\frac{1}{4}$ in. outside diameter × 18 ft $6\frac{1}{2}$ in.) so that the couple could be moved up and down the centre of the tube. In this way, they found that the temperature rose slightly from the bottom of the tube to the point where boiling commenced, after which the change in temperature was relatively small. Applying this technique, it was possible to determine the heat transfer coefficients in the non-boiling and boiling sections of the tube.

## OPERATION OF THE EVAPORATOR

### Heat Transfer Coefficients

The normal rate equation for heat transfer is given in the form,

$$Q = U \,.\, A \,.\, \Delta T \qquad \ldots.(21.1)$$

where $Q$ is the heat transferred per unit time,
$U$ is the overall heat transfer coefficient,
$A$ is the heat transfer surface, and
$\Delta T$ is the temperature difference between the two streams.

In applying this relation to evaporators, there is some difficulty in deciding the correct value for the temperature difference. This arises from what are known as the boiling point rise (B.P.R.) and the hydrostatic head. If water is boiled in an evaporator under a given pressure, then the temperature of the liquor can be determined from steam tables and the temperature difference is readily calculated. At the same pressure, a solution has a boiling point greater than that of water, and the difference between its boiling point and that of water is the B.P.R. For example, at atmospheric pressure, a 25% solution of sodium chloride boils at 226°F and shows a B.P.R. of 14°F. Thus, if steam at 240°F were used to concentrate the salt solution, the overall temperature difference would not be 240 — 212 = 28°F, but only 240 — 226 = 14°F.

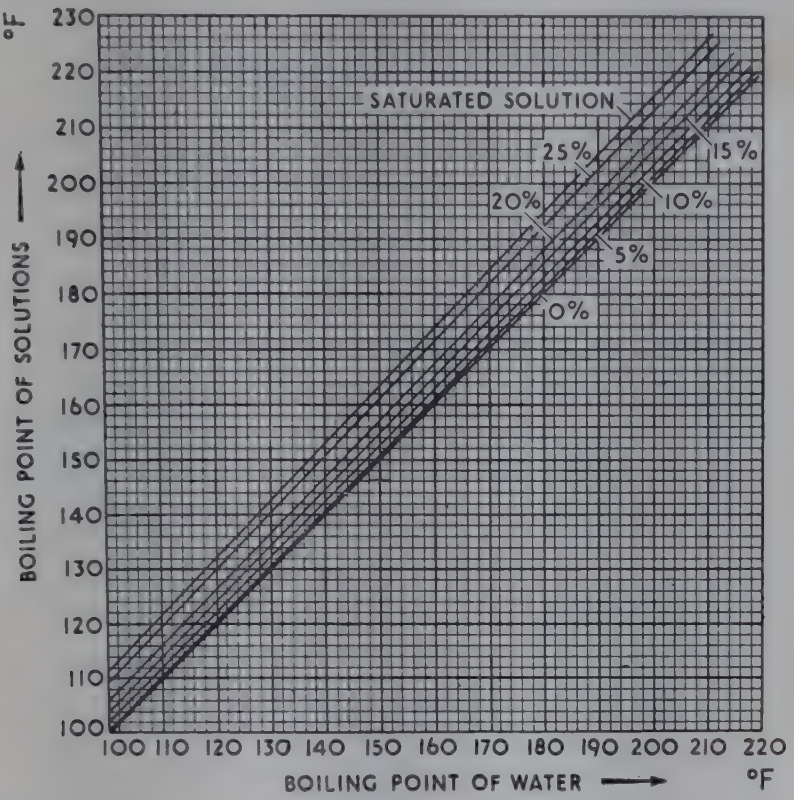

Fig. 21.12. Boiling point of solutions of sodium chloride as a function of the boiling point of water (Dühring lines)

Such solutions usually require more heat to vaporise one pound of water, so that the reduction in capacity of a unit may be considerable. The value of the B.P.R. cannot be calculated from physical data of the liquor, but DÜHRING's rule is often used to find the change in B.P.R. with pressure. If the boiling point of the solution is plotted against that of water at the same pressure, then a straight line is obtained, as shown for sodium chloride in Fig. 21.12. Thus, if the pressure is fixed, the boiling point of water is found from steam tables, and the boiling point of the solution from Fig. 21.12. The boiling point rise is much greater with strong electrolytes, such as salt and caustic soda.

The effect of hydrostatic head may be considered by supposing the liquor to be boiling at the top of a tube. Then the pressure of the liquid which is just at the top of the tubes is that in the vapour space, and the boiling point can therefore be found. The liquor at the bottom of the tubes is at a higher pressure due to the hydrostatic head; and, if the liquor is to boil, it must be heated to the higher temperature corresponding to the increased pressure. Thus, the temperature difference between the steam outside the tubes and the liquor will depend on where boiling starts, and there is no satisfactory way of determining this. In a natural circulation unit with vertical tubes, the liquid flows down the central downtake and enters the bottom of the tubes at a temperature below the boiling point at this depth. On rising, the liquor is heated and at the same time the pressure falls, so that at some point the liquid starts to boil. In calculating the transfer coefficient $U$ from equation 21.1, if the boiling point of the liquor is taken as that of water at that pressure, then the values of $U$ are known as the

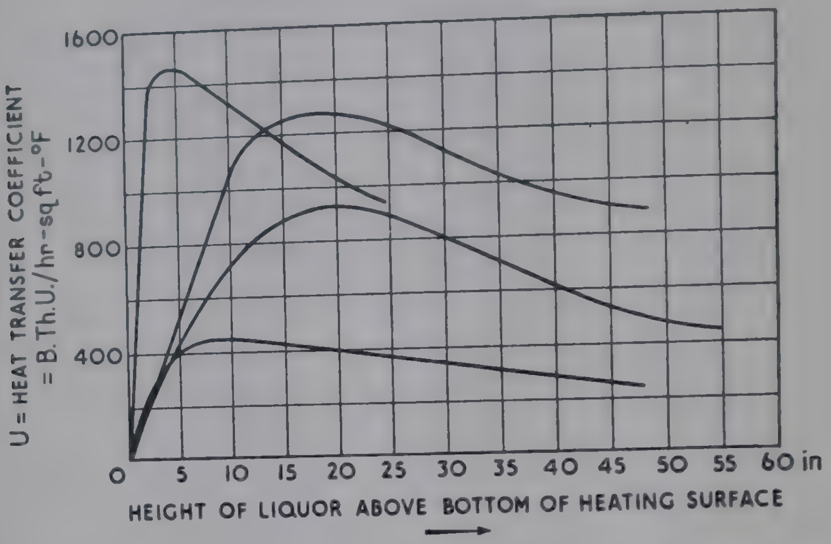

Fig. 21.13. Effect of liquor depth on heat transfer coefficient for various natural circulation evaporators

apparent transfer coefficients. If the boiling point is corrected for B.P.R., the values of $U$ are known as the corrected coefficients. Further correction for the hydrostatic head is not generally attempted. The real temperature difference, and $U$ in equation 21.1, will alter with the depth of liquor. This has been shown by BADGER and SHEPARD(3), and from Fig. 21.13 it is seen that, after an initial sharp rise, $U$ falls as the liquor level is increased. The problem is complicated by the fact that, as the level is reduced, the rate of circulation of the liquor rises.

***Film Transfer Coefficients.**** The overall transfer coefficient for any form of evaporator will depend on the values of the film coefficients on the heating side and for the liquor, together with allowances for scale deposits and the tube metal. For condensing steam, the values obtained in practice are of the order of 1000 B.Th.U./hr-ft²-°F (1000 lb-cal/hr-ft²-°C). There is no entirely satisfactory method of calculating the values for the boiling film but, in a recent analysis of nucleate boiling by MCNELLY(21), the following relation was found to fit the majority of published results with fair accuracy.

$$\frac{h_b d}{k} = 0{\cdot}225 \left[\frac{C_p q d}{k\lambda}\right]^{0{\cdot}69} \left[\frac{P d \rho_L}{\sigma \rho_v}\right]^{0{\cdot}31} \quad \ldots.(21.2)$$

---

* All numerical values of heat transfer coefficients are expressed as B.Th.U./hr-ft²-°F or lb-cal/hr-ft²-°C.

If this condensate is returned to the boiler house, then at least 972 B.Th.U. (540 lb-cal) per pound must be added to give 1 lb of steam to be fed back to the evaporator. In practice, of course, more heat per pound of condensate will be required.

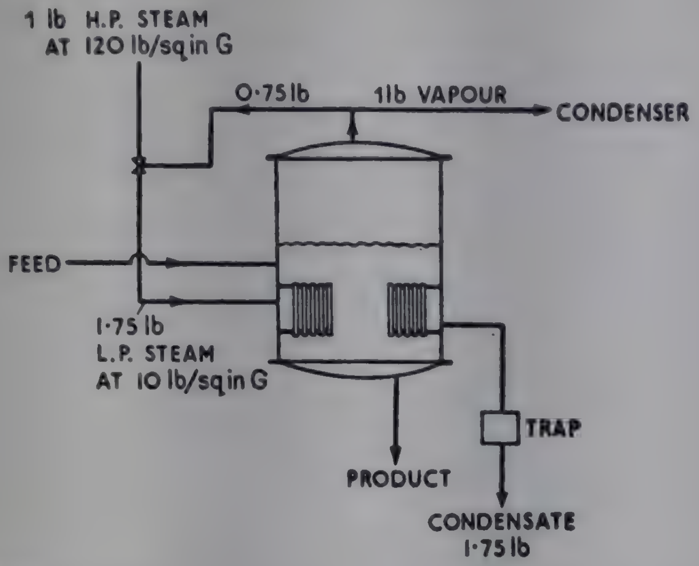
1 lb H.P. STEAM
AT 120 lb/sq in G
0·75 lb
1 lb VAPOUR
CONDENSER
FEED
1·75 lb
L.P. STEAM
AT 10 lb/sq in G
TRAP
PRODUCT
CONDENSATE
1·75 lb

Fig. 21.20. Vapour compression evaporator with high pressure steam-jet compression

972 B.Th.U. (540 lb-cal) will vaporise 1 lb of liquid at atmospheric pressure to give vapour with a total heat of 1150 B.Th.U. (639 lb-cal). To regenerate 1 lb of steam in the original condition from this requires the addition of only 10 B.Th.U. (5 lb-cal). The idea of vapour compression is to make use of the vapour from the evaporator, and to upgrade it to the condition of the original steam. Such an idea offers enormous advantages in thermal economy, but it is by no means easy to add the 10 B.Th.U. (5 lb-cal) to each pound of vapour in an economical manner. The two methods available are:

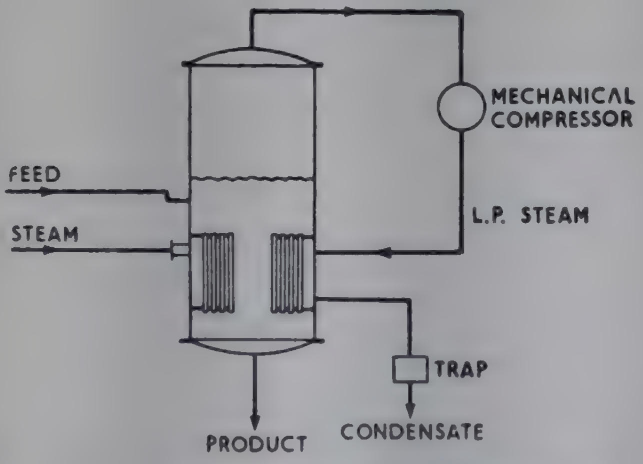
MECHANICAL
COMPRESSOR
FEED
STEAM
L.P. STEAM
TRAP
PRODUCT
CONDENSATE

Fig. 21.21. Vapour compression evaporator with mechanical compressor

(1) The use of steam-jet ejectors (Fig. 21.20).

(2) The use of mechanical compressors (Fig. 21.21).

In selecting a compressor for this type of operation, the main difficulty is the very large volume of vapour to be handled. Rotary compressors of the Rootes type are suitable for small and medium size units, but have not often been applied to large installations. Mechanical compressors have been used extensively for the purification of sea water.

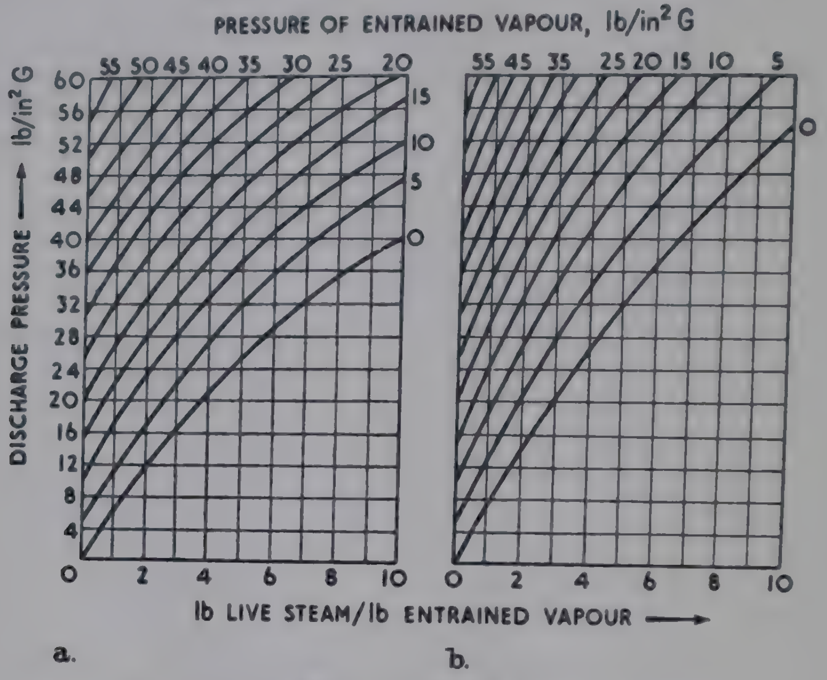

Fig. 21.22. Performance of steam jet ejector
(*a*) 100 lb/in.$^2$ G operating pressure (*b*) 150 lb/in.$^2$ G operating pressure

The use of an ejector, fed with high-pressure steam, is illustrated in Fig. 21.20. High-pressure steam is injected through a nozzle and the low-pressure vapours are drawn in through a second nozzle at right angles, the issuing jet of steam passing out to the calandria, as indicated. These units are relatively simple in construction and can be made of corrosion-resistant material; they have no moving parts and for this reason will have a long life. They have the great advantage over the mechanical compressor that they will handle large volumes of vapour and can therefore be arranged to operate at very low pressures. The disadvantage of the steam jet ejector is that it works at maximum efficiency at only one specific condition. Some indication of the performance of these units is shown in Fig. 21.22, where the pressure of the mixture, for different amounts of vapour compressed per pound of live steam, is shown for a series of different pressures. With an ejector of these characteristics using steam at 125 lb/sq in. G, we can compress 0·75 lb of vapour per pound of steam to give 1·75 lb of vapour at 10 lb/sq in. G. An evaporator unit, as shown in Fig. 21.20, will therefore give 1·75 lb of vapour per pound of high pressure steam. Of the 1·75 lb of vapour, 0·75 lb is taken to the compressor and the remaining 1 lb to the condenser. Ideally, this single effect unit gives an economy of 1·75, or approximately the economy of a double effect unit.

Two illustrations, taken from German industry, illustrate the application of this system. In the first, 4300 Kg/hr of a potato waste liquor are concentrated from 5·5 to 45% solids in a single effect fitted with vapour compression. 2150

Kg/hr of steam at 7 atmospheres are used and 3800 Kg of water are evaporated per hour, corresponding to an economy of 1·7.

In a second installation, a single effect with vapour compression concentrates 376 Kg/hr of a glycerine-water liquor from 5% to 88% glycerine, by the evaporation of 355 Kg of water per hour, and uses 210 Kg/hr of steam at 16 atmospheres. This corresponds to an economy of 1·7.

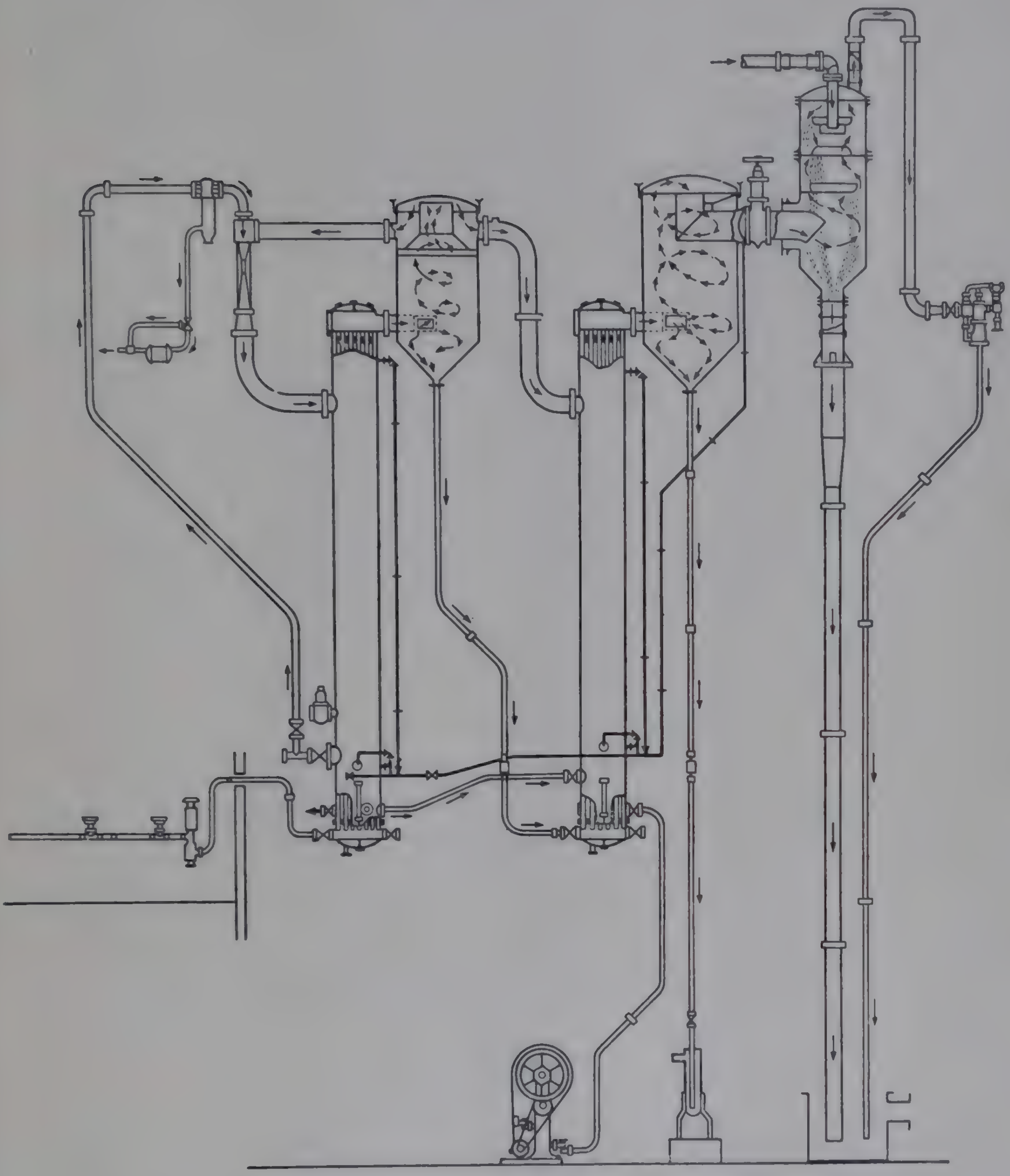

Fig. 21.23. Vapour compression on first effect of two stage evaporator

Vapour compression may be applied to the first effect of a multiple effect system, thus giving increased utilisation of the steam (Fig. 21.23). Such a device is not suitable for use with liquors having a high boiling-point rise, for in these cases the vapour, although initially superheated, has to be compressed to such an extent, in order to give the desired temperature difference across the calandria, that the efficiency is reduced. The application of these compressors is bound up with the steam load of the factory. If there is plenty of low pressure steam available, then the use of vapour compression can scarcely be advocated. If, however, high-pressure steam is available, then it can be used to advantage in a vapour compression unit. It will, in fact, be far superior to the practice of passing high-pressure steam through a reducing valve to feed an evaporator.

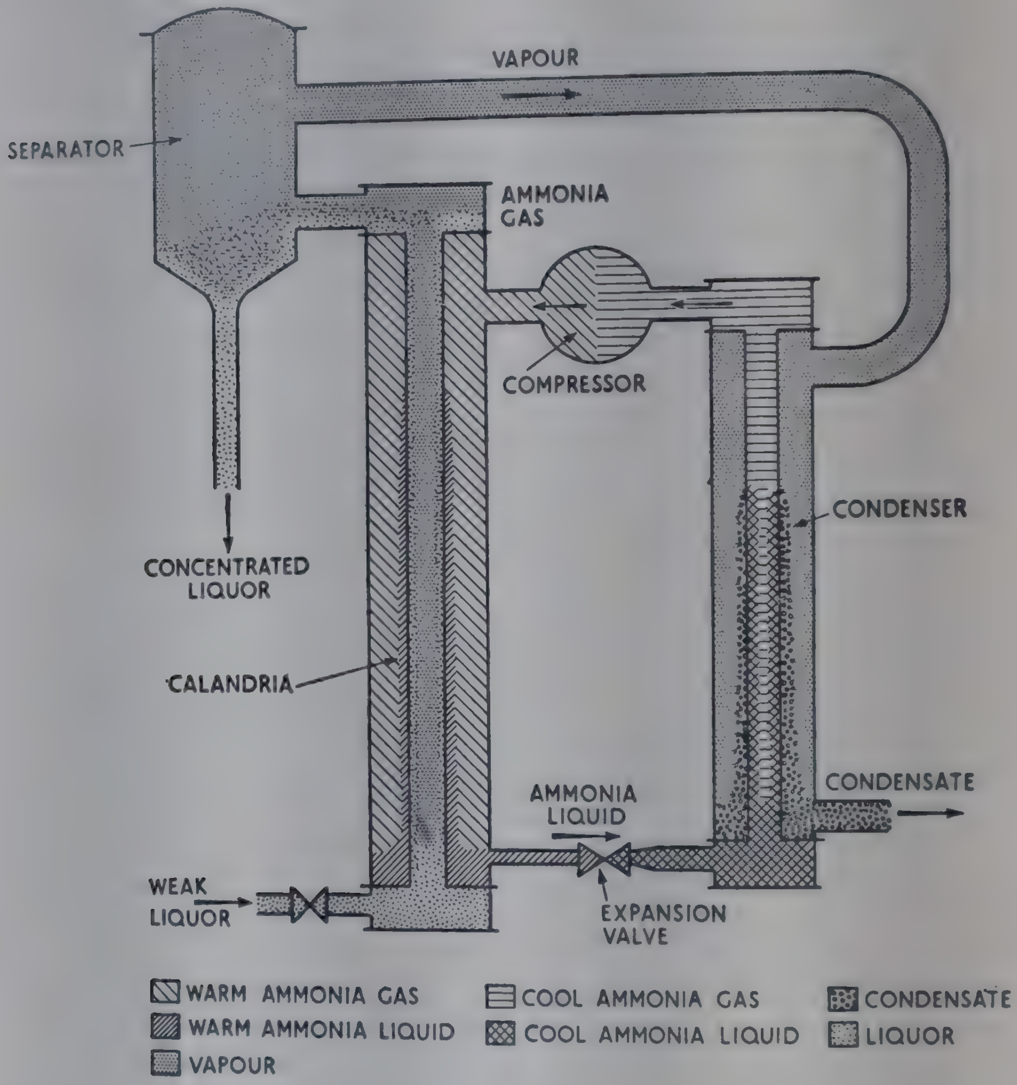
VAPOUR
SEPARATOR
AMMONIA GAS
COMPRESSOR
CONDENSER
CONCENTRATED LIQUOR
CALANDRIA
AMMONIA LIQUID
CONDENSATE
WEAK LIQUOR
EXPANSION VALVE
▧ WARM AMMONIA GAS
▤ COOL AMMONIA GAS
▦ CONDENSATE
▨ WARM AMMONIA LIQUID
▩ COOL AMMONIA LIQUID
▒ LIQUOR
▒ VAPOUR

Fig. 21.24. Heat pump cycle using ammonia

***The Heat Pump Cycle.*** The evaporation of citrus juices at temperatures up to 130°F (55°C), or of pharmaceutical products at even lower temperatures, has led to the development of the evaporator incorporating a heat pump cycle using a separate working fluid. Fig. 21.24 shows the use of the heat pump cycle, with ammonia as the working fluid. In this arrangement, ammonia gas vaporises the feed liquor at a low temperature such as 80–100°F (25–40°C), the ammonia being condensed. The liquid ammonia is then passed through an expansion valve, where it is cooled to a much lower temperature. The cooled ammonia liquor then enters the condenser where it condenses the vapour leaving the separator; it is itself vaporised and leaves as low pressure gas, to be compressed in a mechanical compressor and then passed to the evaporator for a second cycle. The excess heat introduced by the compressor must be removed from the ammonia by means of a cooler.

The main advantage of this form of unit lies in the very great reduction in the volume of gas handled by the compressor. Thus, one pound of water vapour at say 100°F (38°C), with a volume of 350 $\text{ft}^3$ and latent heat about 1100 B.Th.U./lb (611 lb-cal/lb), passes this heat to ammonia at a temperature of say 90°F (32°C). About 2·1 lb of ammonia will be vaporised to give a vapour with a volume of only about 3·5 $\text{ft}^3$ at the high pressure used in the ammonia cycle.

SCHWARZ(17) gives a comparison of the various units used for low temperature evaporation. The three types in general use are the single effect single pass, the single effect with recirculation, and the multiple effect with recirculation. For each of these, we may have vapour compression or the addition of a second heat transfer medium. SCHWARZ suggests that the multiple effect units are the most economical, in terms of capital and operating costs. It is important to note that the single-effect, single-pass system offers the minimum hold-up, and hence a very short transit time. In view of recent work on film-type units, there seems little to be gained by recirculation, since over 70% vaporisation can be achieved in one pass. The figures in Table 21.2 show the comparison between a double-effect unit with vapour compression on the first effect, and a unit with an ammonia refrigeration cycle, both units giving 10,000 lb/hr of evaporation.

TABLE 21.2. *Comparison of refrigeration and vapour compression systems*

| System | Steam lb/hr at 125 lb/sq in. G. | Water gal/min at 80°F (27°C) | Power kW |
|---|---|---|---|
| Refrigeration cycle | 492 | 250 | 320* |
| Vapour compression | 7500 | 1000 | 20 |
| Ratio of steam system to refrigeration | 15·1 | 4 | 0·06 |

* Includes 400 H.P. compressor.

The utilities, other than power, required for the refrigeration system are therefore very much less than for recompression with steam, but the capital cost and the cost of power will be much higher.

Fig. 21.25 shows a double effect system with a capacity of 6000 lb/hr, using an ammonia refrigeration cycle; the first effect is heated by warm water obtained from the ammonia condenser. The first effect has a surface of 1750 $\text{ft}^2$, and the second of 2100 $\text{ft}^2$, the overall transfer coefficients being 75 and 117.

REAVELL(20) has given a comparison of costs for concentration of a feed of a heat sensitive protein liquor at 6 tons per hour from 10% to 50% solids, on the basis of a 160-hour week. It should be noted that, when using the double effect with vapour compression, a lower temperature can be used

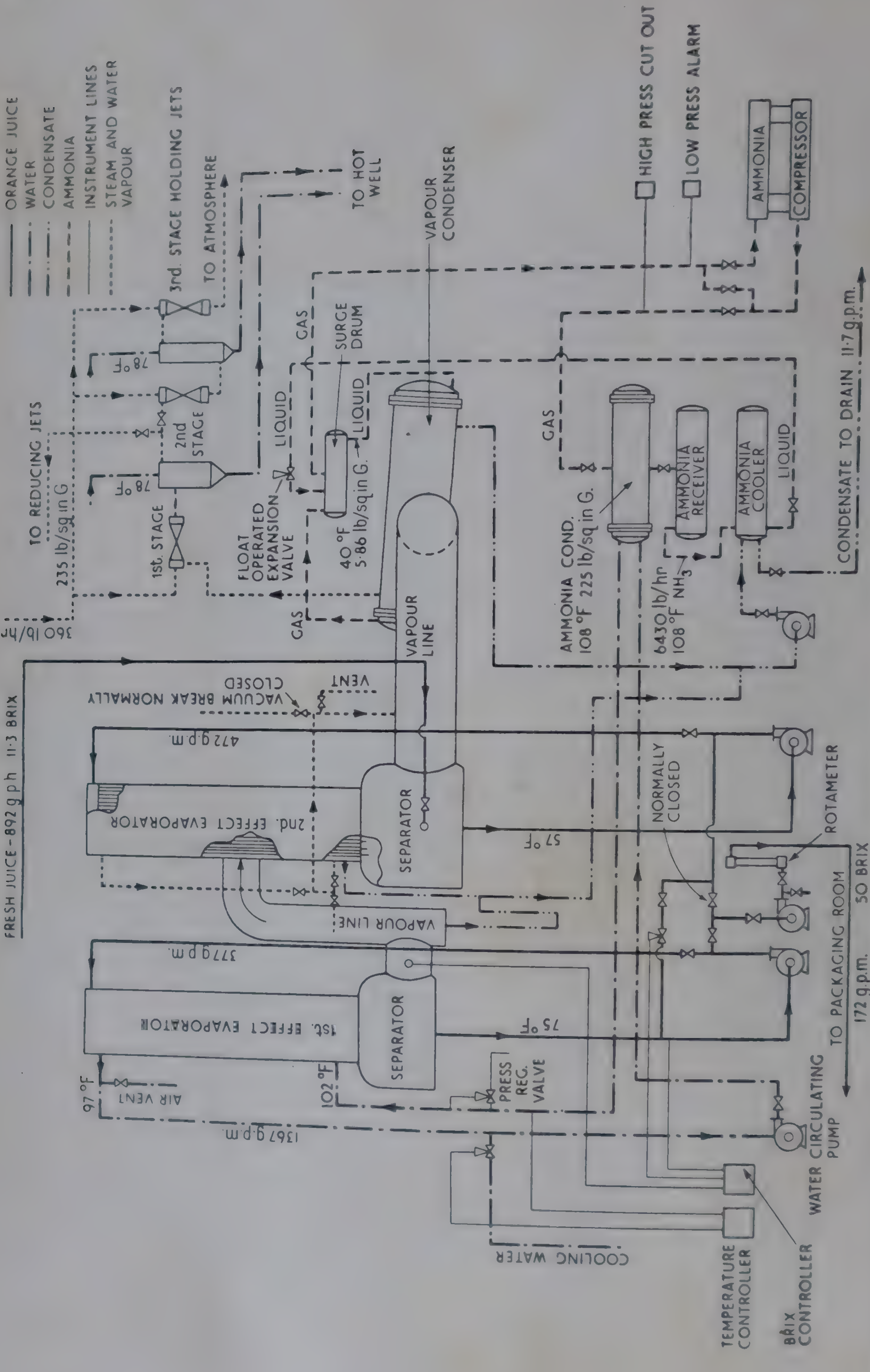
ORANGE JUICE
WATER
CONDENSATE
AMMONIA
INSTRUMENT LINES
STEAM AND WATER VAPOUR
3rd. STAGE HOLDING JETS
TO ATMOSPHERE
TO HOT WELL
VAPOUR CONDENSER
HIGH PRESS CUT OUT
LOW PRESS ALARM
AMMONIA
COMPRESSOR
TO REDUCING JETS
235 lb/sq in G
78°F
78°F
2nd STAGE
1st. STAGE
GAS
SURGE DRUM
LIQUID
LIQUID
FLOAT OPERATED EXPANSION VALVE
40°F
5·86 lb/sq in G.
GAS
AMMONIA COND.
108°F 225 lb/sq in G.
AMMONIA RECEIVER
AMMONIA COOLER
LIQUID
CONDENSATE TO DRAIN 11·7 g.p.m.
360 lb/hr
GAS
VAPOUR LINE
6430 lb/hr
108°F NH$_3$
FRESH JUICE - 892 g p h 11·3 BRIX
VACUUM BREAK NORMALLY CLOSED
VENT
472 g.p.m.
2nd. EFFECT EVAPORATOR
SEPARATOR
57°F
NORMALLY CLOSED
ROTAMETER
VAPOUR LINE
377 g.p.m.
1st. EFFECT EVAPORATOR
SEPARATOR
75°F
TO PACKAGING ROOM
172 g.p.m.
50 BRIX
97°F
AIR VENT
102°F
PRESS REG. VALVE
1367 g.p.m.
WATER CIRCULATING PUMP
COOLING WATER
TEMPERATURE CONTROLLER
BRIX CONTROLLER

Fig. 21.25. Double effect falling-film evaporator with ammonia refrigeration, for orange juice

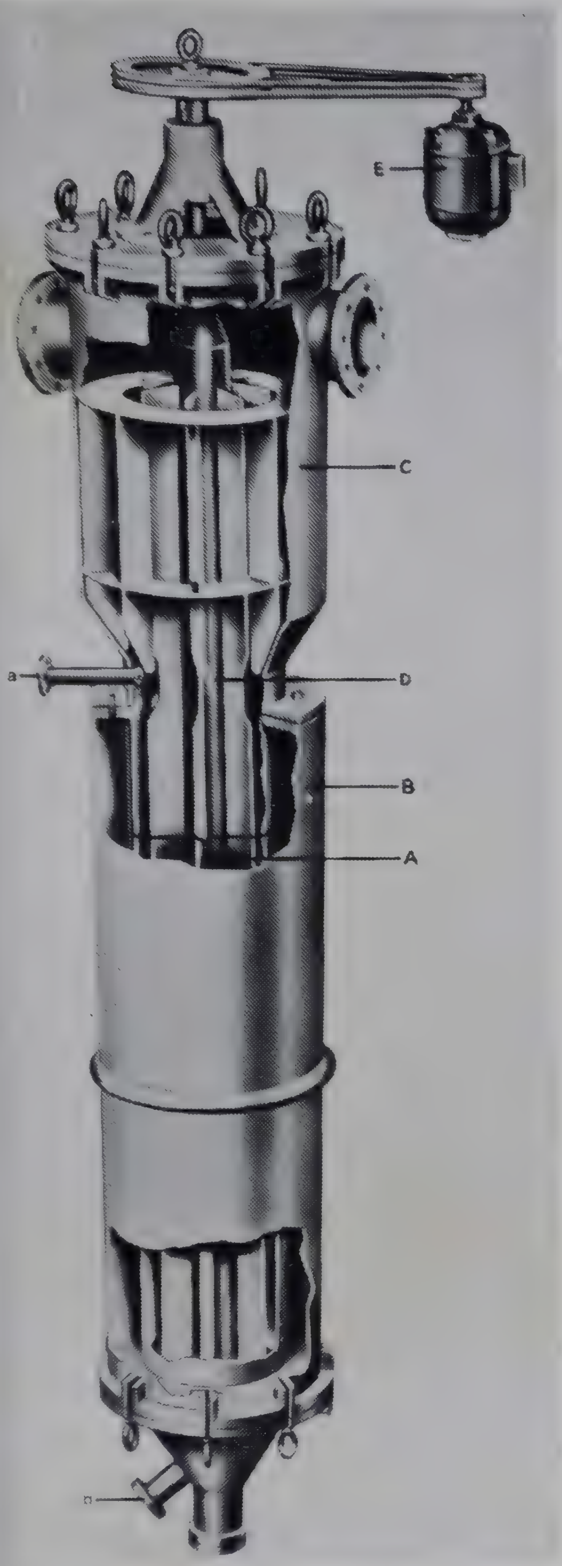

Fig. 21.26. Luwa evaporator working on thin films

*A*—vertical tube
*B*—jacket
*C*—separator
*D*—rotor driven by external motor
*E*—driving motor
*a*—liquid inlet
*b*—liquid outlet

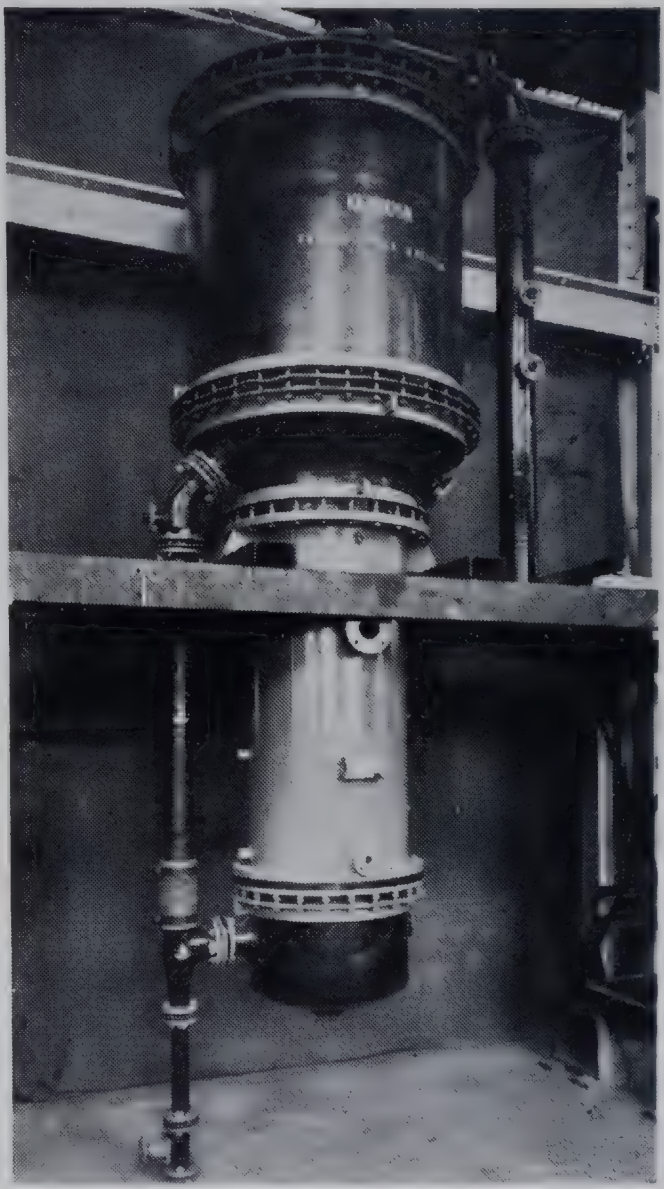

Fig. 21.27. Climbing film evaporator with carbon tubes and Keebush separator

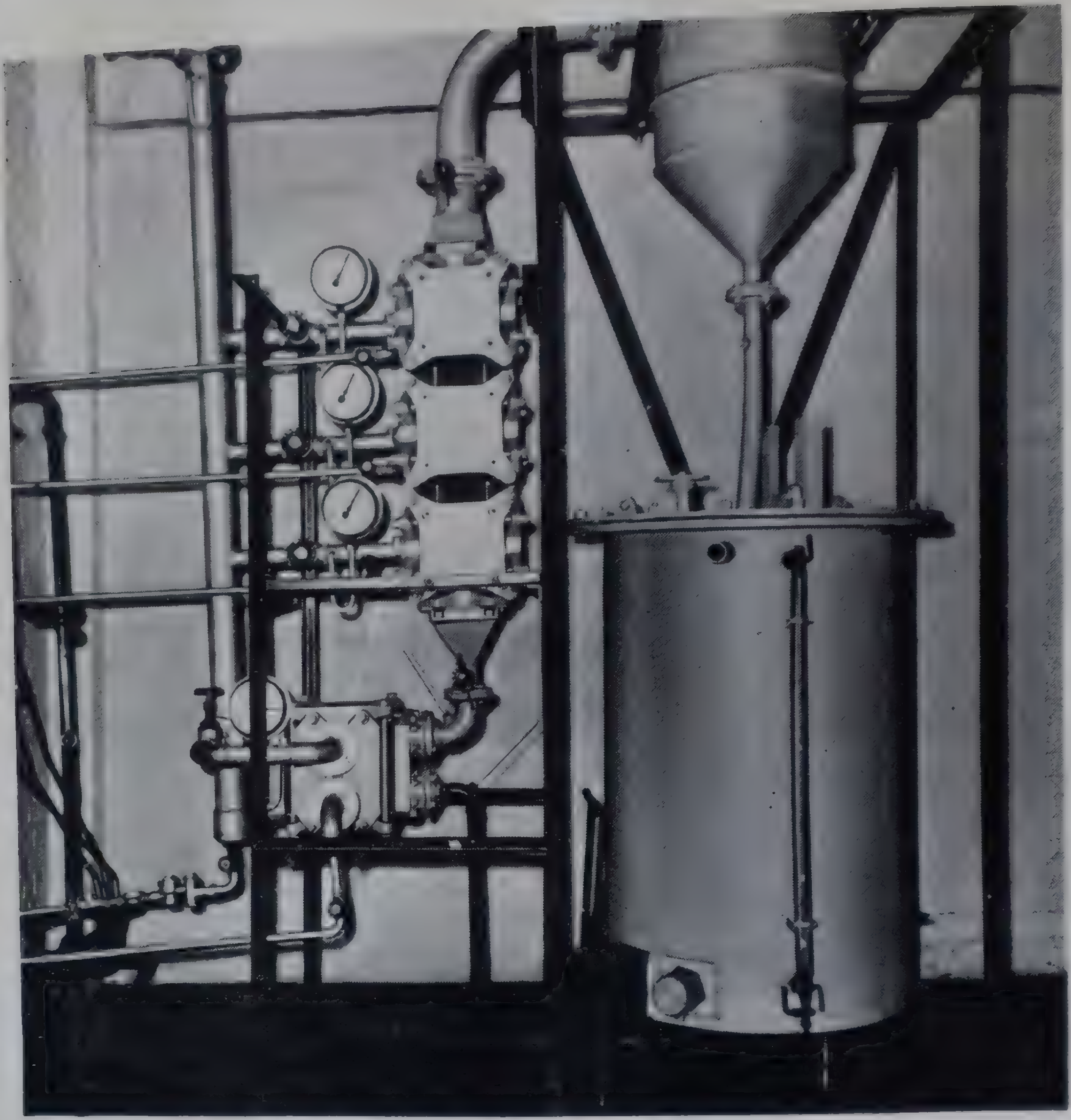

Fig. 21.28. Powell-Duffryn carbon tube evaporatoı

in the first effect than when a triple effect unit is used. In determining these figures no account has been taken of depreciation, but if this is taken as 15% of the capital costs it will not make a significant difference to the comparison.

TABLE 21.3. *Comparison of various systems for concentration of protein liquid*

| Type | Approx. Installed cost | Cost of steam per year | Net saving per year compared with single effect |
|---|---|---|---|
| Single effect | £5,000 | £40,300 | — |
| Double effect | £7,000 | £21,400 | £18,900 |
| Double effect with vapour compression | £9,000 | £13,700 | £26,600 |
| Triple effect | £10,000 | £14,300 | £26,000 |

## SOME NEWER TYPES OF EVAPORATORS

A new form of evaporator in which thin films are used has been developed by LUWA, S. A., of Zurich. As illustrated in Fig. 21.26, it consists of a vertical tube *A*, the lower portion of which is surrounded by a jacket *B* which contains the heating medium. The upper part of the tube *A* is not jacketed and acts as a separator. A rotor *D*, driven by an external motor, has blades which extend nearly to the bottom of the tube, so mounted that there is a small clearance of only about 0·05 in. between their tips and the jacket. The liquor to be concentrated enters at *a* and is picked up by the rotating blades and thrown against the wall of the tube. This action provides a thin film of liquid and sufficient agitation to give good heat transfer, even with very viscous liquids. The film flows down by gravity, becoming concentrated as it falls. The concentrated liquor is taken off from the bottom by a pump, and the vapour leaves at the top of the unit, where it is passed to a condenser. With this form of equipment, the short time of contact of the normal falling-film unit is obtained.

Two recent units, making use of carbon and plastic material for construction, are shown in Figs. 21.27 and 21.28. In the equipment shown in Fig. 21.27, 6-ft tubes of carbon are used with a separator of Keebush. A novel form of construction is adopted by the Powell Duffryn Company, as shown in Fig. 21.28. The tubes are formed from a compact carbon block, and the evaporator has a large transfer surface per unit volume. These units can easily be assembled together to give increased surface. In commercial installations, they have given good values of overall transfer coefficients. Thus, a value of 200 has been obtained for concentrating thick fruit juice containing syrup, and also for concentrating 40% sulphuric acid to 60%. A somewhat lower figure of 140 is obtained for sulphuric acid concentration from 60 to 74% at a pressure of 11 mm of mercury, and similar values are obtained with spinning-bath liquors and some pharmaceuticals.

## SEPARATORS AND ENTRAINMENT

In operating an evaporator, it is important to minimise entrainment of the liquid in the vapour passing over to the condenser. Entrainment is reduced by having a considerable headroom, of say 6 ft, above the boiling liquid, but the addition of some form of de-entrainer is usually essential. Fig. 21.29 shows three methods of reducing entrainment. The simplest is to take the vapour from an upturned pipe as in *a*, and this has been found to give quite good results in small units. The deflector type *b* is a common form of de-entrainer and the tangential separator *c* is the type usually fitted to climbing-film units. This problem is particularly important in the concentration of radioactive waste liquors and has been discussed by McCullough[18]. He cites the case of a

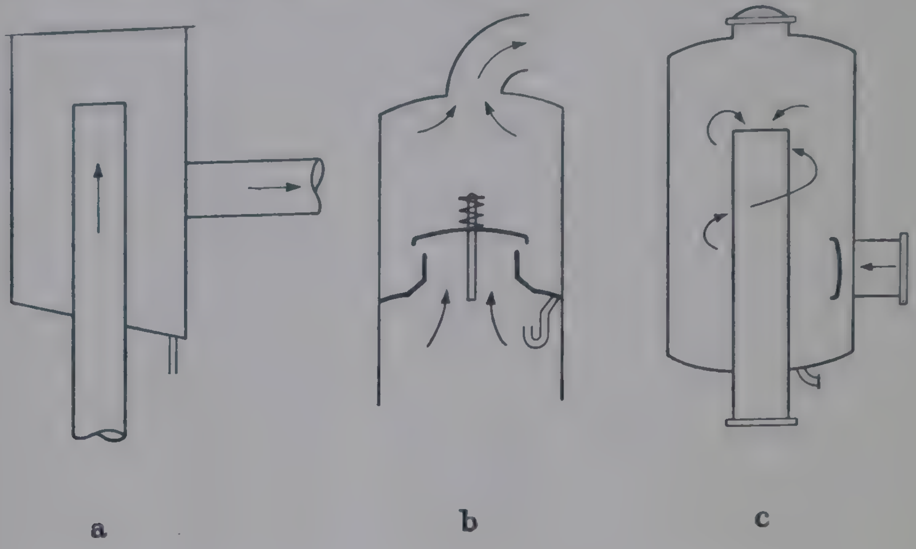

Fig. 21.29. Entrainment separators
(*a*) Upturned pipe (*b*) Deflector type (*c*) Tangential type

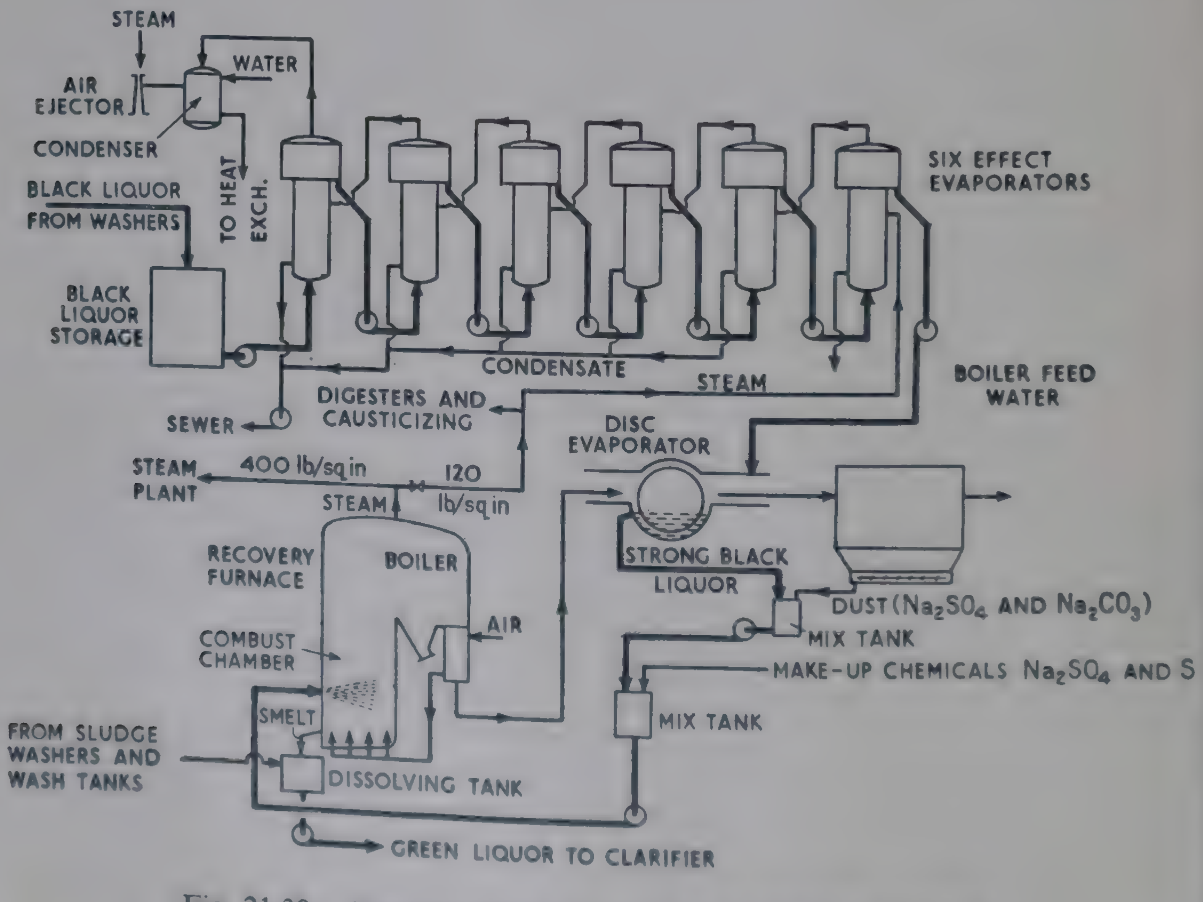
STEAM
WATER
AIR EJECTOR
CONDENSER
BLACK LIQUOR FROM WASHERS
TO HEAT EXCH.
SIX EFFECT EVAPORATORS
BLACK LIQUOR STORAGE
CONDENSATE
STEAM
BOILER FEED WATER
SEWER
DIGESTERS AND CAUSTICIZING
DISC EVAPORATOR
STEAM PLANT
400 lb/sq in
120 lb/sq in
STEAM
RECOVERY FURNACE
BOILER
STRONG BLACK LIQUOR
DUST ($Na_2SO_4$ AND $Na_2CO_3$)
AIR
MIX TANK
COMBUST CHAMBER
MAKE-UP CHEMICALS $Na_2SO_4$ AND S
SMELT
MIX TANK
FROM SLUDGE WASHERS AND WASH TANKS
DISSOLVING TANK
GREEN LIQUOR TO CLARIFIER

Fig. 21.30. Sextuple effect evaporator for the paper industry

batch evaporator of the forced circulation type in which the vapours are passed to a 12 ft diameter separator and then through four bubble cap trays to give complete elimination of entrained liquor. A good entrainment separator will reduce the amount of liquid carried over to between 10 and 20 lb per $10^6$ lb of vapour.

## SOME INDUSTRIAL INSTALLATIONS

An installation for the concentration of black liquor in the Kraft paper industry of America has been described by SAWYER(10). The main evaporating unit, shown in Fig. 21.30, consists of a sextuple-effect system using long tube evaporators, each evaporator containing 552 tubes of 2-in. internal diameter and 24 ft long in a steam shell of 4 ft diameter with a dome of 8 ft diameter. The material and heat balances for this unit are given in Table 21.4. The liquor leaving this unit is too viscous for further concentration in the film

TABLE 21.4. *Sextuple-effect Evaporator Test*

Material Balance (lb/hr)

| | | |
|---|---|---|
| Feed of weak black liquor | = 2850 cu ft/hr × 1·09 (sp gr) × 62·4 lb/cu ft | 194,000 |
| Discharge from evaporator | $= 194{,}000\ \text{lb/hr} \times \dfrac{17{\cdot}5\ (\%\ \text{solids in inlet liquor})}{53{\cdot}3\ (\%\ \text{solids in discharge liquor})}$ | 63,700 |
| Total evaporation of water accomplished in evaporator | | 130,300 |

$$\text{Lb } H_2O \text{ evaporated/lb steam} = \frac{130{,}300\ \text{lb/hr (total evaporation)}}{26{,}000\ \text{lb/hr (steam discharge liquor)}} = 5{\cdot}01.$$

| | Flash Tank | Evaporator Test Effect[a] | | | | | |
|---|---|---|---|---|---|---|---|
| | | I | II | III | IV | V | VI |
| Chest pressure lb/sqin. G | — | 36·5 | 15·0 | 9·2 | −4·0[b] | −15·0[b] | −21·2[b] |
| Chest temperature, °F | — | 282 | 249 | 226 | 205 | 179 | 156 |
| Temp. diff., °F | — | 21 | 12 | 12 | 14 | 11 | 31 |
| Liquor temp. calcd., °F | 217 | 261 | 237 | 214 | 191 | 168 | 125 |
| Liquor temp. measured, °F | 206 | 263 | 234 | 210 | 186 | 164 | 129 |
| Boiling point rise | 11 | 11 | 10 | 8 | 7 | 5 | 6 |
| Dome temperature, °F | 206 | 250 | 227 | 206 | 181 | 163 | 119 |
| Dome pressure, in. Hg | — | 30·8 | 10·9 | −3·5 | −13·3 | −19·6 | −26·7 |
| Latent heat of vaporisation B.Th.U./lb | 974 | 946 | 961 | 974 | 990 | 1000 | 1026 |
| Total solids, % | 53·3 | | 37·9 | 31·3 | 25·9 | 22·4 | 24·3 |
| Feed, lb/hr | 66,000 | 90,000 | 108,500 | 131,000 | 145,300 | 95,300 | 98,700 |
| Discharge lb/hr | 63,700 | 66,000 | 90,000 | 108,000 | 131,000 | 74,300 | 71,000 |
| Evaporation, lb/hr | 2,300 | 24,000 | 18,500 | 23,500 | 14.300 | 21,000 | 27,700 |
| Evaporation (heat bal.) lb/hr | 2,240 | 23,500 | 21,700 | 22,100 | 19,800 | 22,300 | 28,500 |
| Over-all temp. diff. % | — | 20·8 | 11·9 | 11·9 | 13·8 | 10·9 | 30·7 |

[a] Total temperature difference in 6 effects = 101°F; inlet weak black liquor temperature = 178°F; barometric pressure = 30·05 in. Hg.

[b] Inches of mercury.

evaporator, and is further treated in two large disc units each $8\frac{1}{2}$ ft in diameter with 33 discs spaced $2\frac{5}{8}$ in. apart. These discs pick up the liquor and evaporation is effected by passing hot gases at 380°F (195°C) over the discs. This arrangement gives a final concentration of 64% solids. A combination of the long tube unit and the direct contact system is a method of making use of the best features of the two types. The backward feed system adopted again has technical advantages in this case.

A new installation for the concentration of brine has recently been installed at the works of the Murgatroyd Vacuum Salt Company$^{(12)}$. The brine, after purification to remove the calcium and magnesium salts, passes through a triple effect vertical evaporator with parallel feed. The units are 11 ft in diameter, and are of cast iron construction; they are fitted with a draw-off leg for the slurry at the bottom. Part of the brine feed is introduced at the bottom of this leg to wash the crystals free from mother liquor, and the remainder enters immediately below the calandria. This consists of a tube bundle of 1389 copper tubes (6 ft 5 in. long by 2 in. outside diameter), with a central downtake of 3 ft diameter, and has a surface area of 3300 sq ft. The natural circulation is augmented by an internal agitator driven from the base, and heat transfer coefficients of 550, 350 and 250 are obtained for the various effects. The slurry drawn from the units is pumped to rotary drum filters, and the filtrate is returned to the evaporators.

## CONDENSING AND VACUUM EQUIPMENT

The vapour leaving the last effect of a multiple effect unit must be condensed, either by direct contact with a jet of water, or in a normal tubular exchanger. If $M$ is the mass of cooling water used per unit mass of vapour in a jet condenser, and $H$ is the enthalpy per unit mass of vapour, a heat balance gives

$$\underset{\text{(Heat in)}}{H + MT_i} = \underset{\text{(Heat out)}}{T_e + MT_e} \qquad \ldots.(21.10)$$

where $T_i$ and $T_e$ are the inlet and outlet temperatures of the water, reckoned above a standard datum temperature, and where the condensate is assumed to leave at the same temperature as the cooling water. From equation 21.10,

$$M = \frac{H - T_e}{T_e - T_i} \qquad \ldots.(21.11)$$

If $T_e$ is 110°F (43°C), $T_i$ is 85°F (29°C), and the vacuum is 26 in. Hg, based on a 30 in. barometer,

$$M = \frac{1116 - 78}{25}\left(\frac{620 - 43}{14}\right) = 41{\cdot}5.$$

The water is then conveniently discharged at atmospheric pressure, without the aid of a pump, by allowing it to flow down a vertical pipe, known as a barometric leg, of sufficient length for the pressure at the bottom to be slightly in excess of atmospheric pressure. (See page 442.) For a jet condenser with a barometric leg, a chart for determining the water requirement has been prepared by ARROWSMITH$^{(9a)}$, and is shown in Fig. 21.31.

### Jet Condensers

Jet condensers may be either of the countercurrent or parallel flow type. In the countercurrent unit, the water leaves at the bottom through a barometric leg, and any entrained gases leave at the top. This provides what is known as a dry vacuum system, since the pump has to handle only the non-condensable gases.

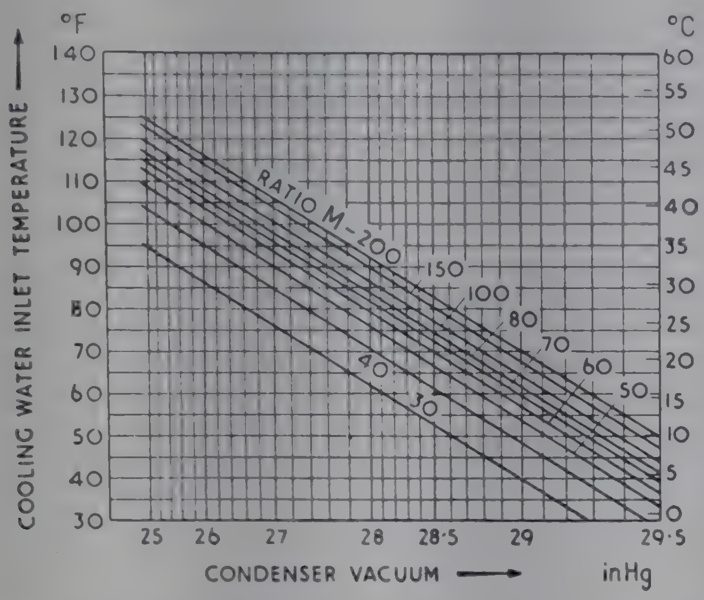

Fig. 21.31. Ratio ($M$) of cooling water to vapour required under various conditions

The cooling water will generally be heated to within 5 or 10°F (3 or 6°C) of the vapour temperature. With the parallel flow system, the temperature difference will be rather greater and, therefore, more cooling water will be required. In this case, the water and gas will be withdrawn from the condenser and passed through a wet vacuum system. As there is no barometric leg, the unit can be mounted at floor level, but the pump displacement is about one and a half times that for the dry vacuum system.

### Vacuum Equipment

Air is introduced into a jet condenser from the cooling water, as a result of the evolution of non-condensable gases in the evaporator, and as a result of leakages. The volume of air to be removed is frequently about 15% of that of the cooling water. The most convenient way of obtaining a vacuum is usually by means of a steam jet ejector (Fig. 21.32). Part of the momentum of a high velocity steam jet is transferred to the gas entering the ejector, and the mixture is then compressed in the diverging portion of the ejector by conversion of kinetic energy into pressure energy. *C* represents the converging-diverging steam nozzle, giving rise to a high velocity jet of steam. In the converging section *B*, this steam entrains gas and, in the diffuser section *D*, part of the kinetic energy of the mixture is converted into pressure energy. Good performance by the ejector is obtained largely by correct proportioning of the steam nozzle and diffuser, and poor ejectors will use much more high pressure steam than a well designed unit. The amount of steam required increases with the compression ratio. Thus, a single-stage ejector will remove air from a system at a vacuum of 25 in. Hg (5 in. absolute pressure); i.e. where a compression ratio of 6 is required. To remove air from a system at 29 in. vacuum would involve a compression ratio of 30, and a single-stage unit would be uneconomic in steam consumption. A two-stage ejector is shown in Fig. 21.33; the first stage withdraws air from the high vacuum vessel and compresses it to say 6 in. absolute pressure, and the second stage compresses the discharge from the first ejector to atmospheric pressure. A further improvement is obtained if a condenser is inserted after the first stage, as

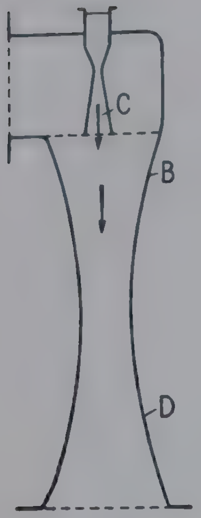

Fig. 21.32.
Steam jet ejector

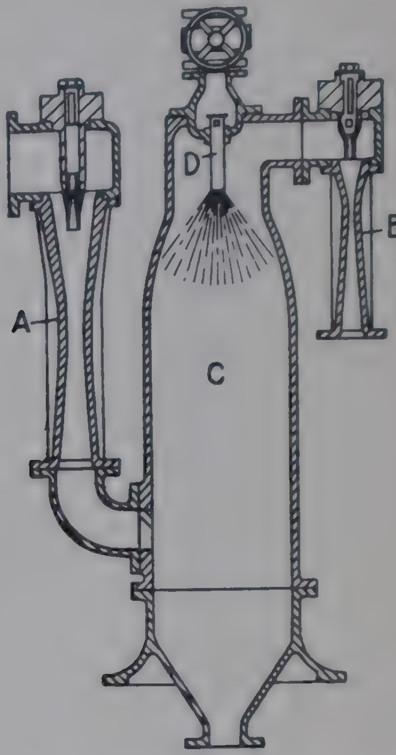

Fig. 21.33. Two-stage ejector with condenser
A—First stage B—Second stage
C—Condenser D—Water spray

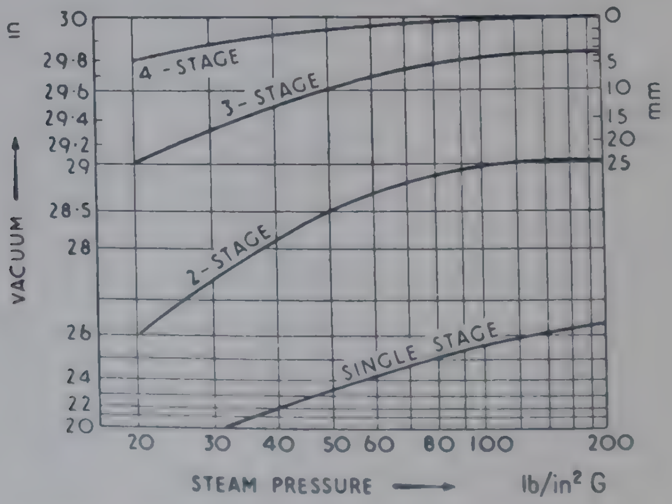

Fig. 21.34. Recommended number of stages for various operating conditions

this will reduce the amount of vapour to be handled in the final stage. This is shown in Fig. 21.33. An indication of the number of stages required for various conditions is shown in Fig. 21.34[(9a)]. The higher the steam pressure the smaller is the consumption, and pressures of 100 to 150 lb/sq. in. G are commonly used in multistage units. Typical performance curves are shown in Fig. 21.35[(9a)],

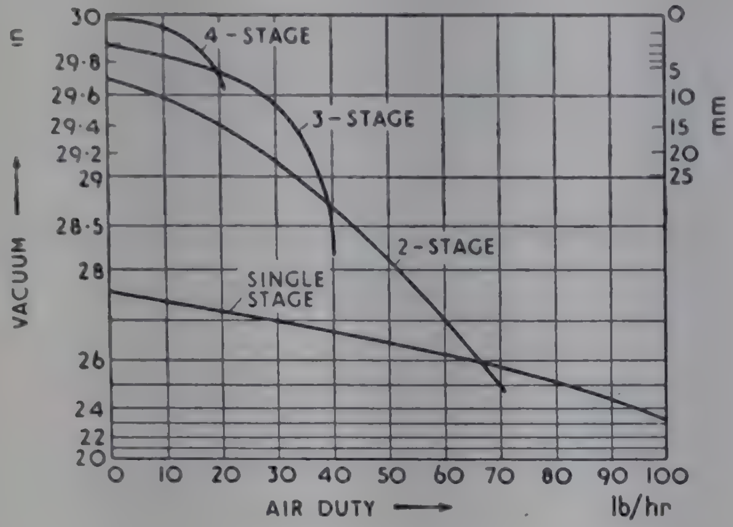

Fig. 21.35. Air duty of steam jet ejectors, for given steam consumption and steam pressure

where the air duty for a given steam consumption and given steam pressure is shown as a function of the number of stages and the operating pressure.

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## LIST OF SYMBOLS USED IN CHAPTER 21

| | | |
|---|---|---|
| $A$ | Heat transfer surface | $\mathbf{L^2}$ |
| $C_p$ | Specific heat of liquid at constant pressure | — |
| $D$ | Liquid evaporated or steam condensed per unit time | $\mathbf{MT^{-1}}$ |
| $d$ | A characteristic dimension | $\mathbf{L}$ |
| $e$ | Economy | — |
| $H$ | Enthalpy per unit mass of vapour | $\boldsymbol{\theta}$ |
| $h_b$ | Film heat transfer coefficient for boiling liquid | $\mathbf{ML^{-2}T^{-1}}$ |
| $k$ | Thermal conductivity of liquid | $\mathbf{ML^{-1}T^{-1}}$ |
| $M$ | Mass of cooling water per unit mass of vapour | — |
| $N$ | Number of effects | — |
| $P$ | Pressure | $\mathbf{ML^{-1}T^{-2}}$ |
| $Q$ | Heat transferred per unit time | $\mathbf{MT^{-1}}\boldsymbol{\theta}$ |
| $q$ | Heat flux | $\mathbf{ML^{-2}T^{-1}}\boldsymbol{\theta}$ |
| $T$ | Temperature | $\boldsymbol{\theta}$ |
| $T_f$ | Feed temperature | $\boldsymbol{\theta}$ |
| $\Delta T$ | Temperature difference | $\boldsymbol{\theta}$ |
| $U$ | Overall heat transfer coefficient | $\mathbf{ML^{-2}T^{-1}}$ |
| $W$ | Feed rate | $\mathbf{MT^{-1}}$ |
| $Z$ | Hydrostatic head | $\mathbf{L}$ |
| $\lambda$ | Latent heat of vaporisation | $\boldsymbol{\theta}$ |
| $\mu$ | Viscosity of liquid | $\mathbf{ML^{-1}T^{-1}}$ |
| $\rho_L$ | Density of liquid | $\mathbf{ML^{-3}}$ |
| $\rho_V$ | Density of vapour | $\mathbf{ML^{-3}}$ |
| $\sigma$ | Interfacial tension | $\mathbf{MT^{-2}}$ |
| *Nu.* | Nusselt number | — |
| *Pr.* | Prandtl number | — |
| *Re.* | Reynolds number | — |

Suffixes 1, 2, 3 refer to the first, second and third effects: 0 refers to the steam side of the first effect; *i* and *e* refer to inlet and outlet cooling water.

## CHAPTER 22

# *Crystallisation*

THE importance of crystallisation in the chemical industry arises very largely from the fact that it is generally more convenient to handle solid particles as opposed to a solution. In a crystal the constituent molecules, ions or atoms are arranged in a regular manner with the result that the crystal shape is independent of size, and if a crystal grows, each of the faces develops in a regular manner. The presence of impurities will, however, usually result in the formation of an irregular crystal. Generally large regular crystals are a guarantee of the purity of the material, though a number of pairs of materials form "mixed crystals."

Crystallisation is normally carried out either from a solution or from a melt, but occasionally crystals are formed directly by condensation from the vapour phase. Many features of the process of condensation from a vapour to a liquid are also common to crystallisation, but only crystallisation is considered here. In some respects crystallisation can be regarded as the inverse of dissolution, which has already been discussed in Chapter 17, but there are important differences. Thus the number of particles present during dissolution will remain constant or decrease, whereas in crystallisation the number of nuclei on which material is deposited may continuously increase. Further, whereas in dissolution there is rarely an appreciable resistance to the transfer of material across the interface between the two phases, this is no longer true for crystallisation.

The crystallisation process consists essentially of two stages which generally proceed simultaneously but which can to some extent be independently controlled. The first stage is the formation of small particles or nuclei, and the second stage is the growth of the nuclei. If the number of nuclei can be controlled the size of the crystals ultimately formed can be regulated, and this forms one of the most important features of the crystallisation process.

### Saturation

If a melt is slowly cooled, the temperature will gradually fall until the melting point is reached and further cooling will then produce, either supercooling of the liquid, or solidification with the temperature remaining approximately constant. Generally a definite degree of supercooling must be achieved before change of phase will occur, and a metastable condition therefore exists at temperatures a little below the melting point because the rate of spontaneous nucleation is here negligible. On the other hand, if a small seed crystal or other nucleus is introduced, it will increase in size until the solution is no longer supercooled.

Similarly if a superheated vapour is cooled, its temperature will fall until it reaches the dry and saturated condition, and further cooling in the absence of suitable condensation nuclei will produce a supersaturated vapour, and condensation of liquid or solid will occur only after a finite degree of supersaturation has been achieved. Fig. 22.1 shows the pressure-temperature diagram for a material, the line *AB* representing the vapour pressure as a function of temperature. If a vapour is cooled from *a* to *c*, it has a supersaturation *bc* with respect to temperature. Condition *c* can also be reached by increasing the pressure, at constant temperature, from *e* to *c*, in which case the supersaturation, in terms of pressure, is *cf*. A vapour can thus become supersaturated by increasing the pressure, by decreasing the temperature, or by both simultaneously. The boundary of the metastable region in which the spontaneous nucleation rate is negligible is represented by the curve *CD* which is roughly parallel to *AB*.

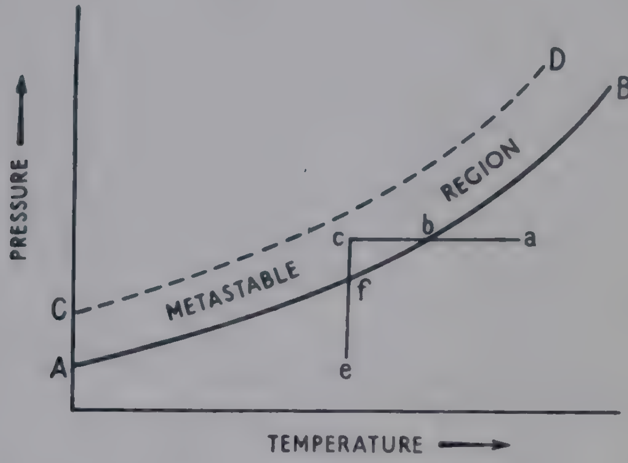

Fig. 22.1. Pressure-temperature relation for saturated vapour

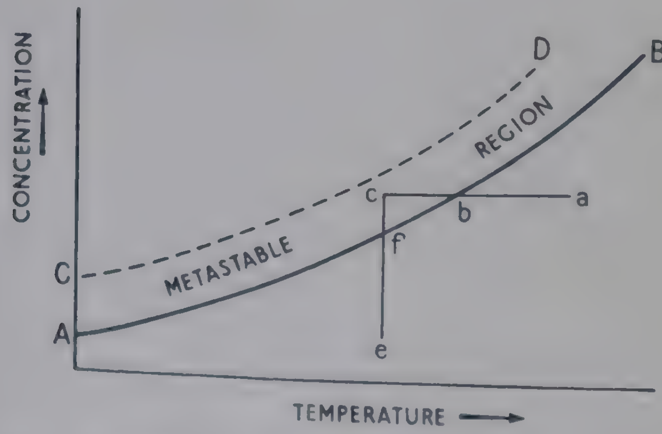

Fig. 22.2. Concentration-temperature relation for saturated solution

Again for a solution, curve *AB* in Fig. 22.2 is the solubility curve and the supersaturation of a solution represented by *c* is *cb* in terms of temperature and *cf* in terms of concentration. *CD* again represents the limit of the metastable region, in which the nucleation rate is very low. It was originally suggested by MIERS(5, 8) and others that there was no nucleation in the metastable region but this is now known to be untrue, and the line *CD* therefore does not represent a sharp boundary.

## Nucleation

Nuclei may form spontaneously if conditions are suitable, but in many cases small seed crystals may be added and small quantities of impurities may also act as nuclei. Spontaneous nucleation is a process which takes place with some reluctance and is thought to depend upon the existence of random variations in concentration or temperature on the molecular scale. LORD KELVIN has shown that the vapour pressure at a convex surface is greater than that at a plane surface under otherwise similar conditions, and is given as a first approximation by

$$P_r = P_s + \frac{2\tau}{r} \cdot \frac{\rho_V}{\rho_L} \qquad \ldots .(22.1)$$

(cf. equation 6.74)

where $P_r$ is the vapour pressure at a convex surface of radius $r$,
$P_s$ is the vapour pressure at a plane surface,
$\tau$ is the interfacial tension, and
$\rho_V$, $\rho_L$ are the densities of the vapour and liquid.

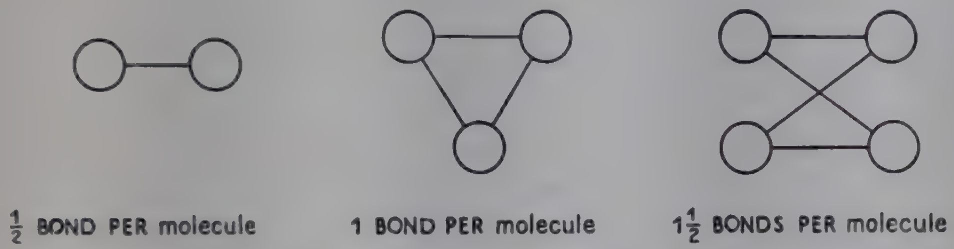

Fig. 22.3. Groups of molecules

This increase in vapour pressure occurs because the molecules in a large droplet are more firmly bound together than those in a small droplet, as can be seen in Fig. 22.3. Thus small droplets may re-evaporate in a slightly supersaturated vapour, and consequently large droplets will tend to grow at the expense of the smaller ones. Although this implies that very small droplets can never form, there are generally sufficiently large irregularities on the molecular scale for the production of a number of nuclei, sufficiently large to be stable. A similar situation arises in the case of a solution where the small particles have a greater solubility than the larger ones, and as a result a nucleus will grow only if it is sufficiently large for the solution to be supersaturated with respect to it.

Study of the nucleation process is in most cases difficult because nucleation and growth usually occur simultaneously. TAMMANN⁽¹⁾ however worked on the crystallisation of a number of somewhat complex organic compounds, such as piperine, from the melt and obtained conditions where the nucleation and growth could be studied virtually independently. Piperine is a compound with a relatively large molecule and melts at 130°C, but nucleation is appreciable only at temperatures below about 100°C. The material was melted in a small tube a few millimetres in diameter and then rapidly cooled to the required temperature for 10 minutes, during which time a finite number of nuclei were formed. The temperature was then rapidly raised to 100°C for 4 minutes to allow them to grow sufficiently to be counted. By this means the curve shown in Fig. 22.4*a* was obtained for the rate of nucleation as a function of temperature. The shape of this curve is readily explained qualitatively. As the temperature is reduced, the degree of supersaturation and the driving force causing nucleation are increased, and the curve rises steeply. Since, however, the kinetics of the process are adversely affected by lowering the temperature, the rate of nucleation attains a maximum value and then falls off as the temperature is further reduced. The effect of temperature on the kinetics of the process is more marked over the period of crystal growth than during nucleation, and the crystallisation rate reaches a maximum at a rather higher temperature than the nucleation. Fig. 22.4*b* shows the effect of temperature on the linear rate of growth of crystals of triphenylguanidine. Whereas the rates of nucleation and of crystallisation can sometimes be studied separately for crystal growth from a melt, this is not possible in the case of a solution because the two rate curves overlap and the effects occur over a much narrower temperature range. It is thought, however, that the general picture is similar.

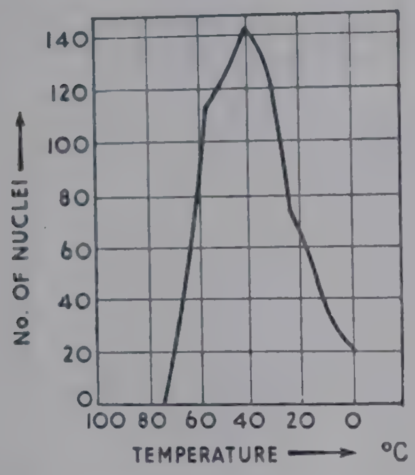

Fig. 22.4*a*. Formation of nuclei from 1·2 cu cm of piperine in 10 minutes (TAMMANN)

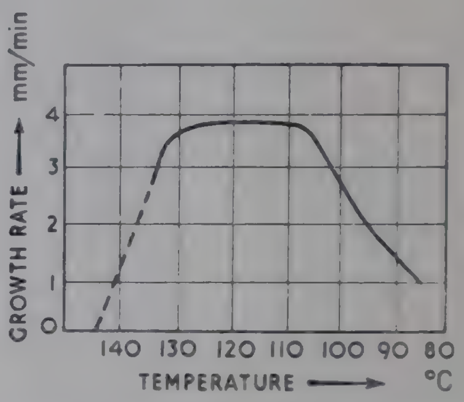

Fig. 22.4*b*. Growth rate of triphenylguanidine crystals (TAMMANN)

The general conclusion is that relatively large crystals will be obtained as a result of slow cooling, because the spontaneous nucleation is then reduced and the material is deposited on a relatively few nuclei. This is borne out in practice. Conversely, rapid cooling produces a large crop of small crystals. Whereas with some materials nuclei form almost immediately supersaturation is achieved, others can be cooled without the deposition of an appreciable number of nuclei, so that crystallisation does not take place and the liquid gradually thickens to form a very viscous mass known as a glass as the temperature is reduced. Sugar syrup and glass are the most commonly encountered glasses, but many organic compounds with large molecules also form glasses very readily. That a glass is a liquid is shown by the fact that it has no crystal structure and that it can flow very gradually.

## Crystallisation Rate

The rate of growth of a crystal in a solution is dependent on the temperature and concentration of the liquid at the crystal face. These conditions are not generally the same as those in the bulk of the solution because a concentration gradient is necessary for the transfer of solute towards the face, and a temperature gradient for the dissipation of the heat of crystallisation. The problem thus involves both heat transfer and mass transfer, though in most cases the heat transfer may be negligible. In the case of melts, the problem is one of heat transfer alone. Since the resistance to heat and mass transfer lies predominantly in the laminar sub-layer close to the surface of the crystal, the rate of growth of the crystal is improved by increasing the relative velocity between the solid and the liquid. Fig. 22.5 shows the effect of increasing the speed of rotation of a crystal of sodium thiosulphate in an aqueous solution. It will be noted that the crystallisation rate initially rises very rapidly but a point is reached where further increase in the agitation produces no effect on the rate of growth. Had the only resistance been within the liquid, the transfer rate would have continued to increase; it therefore appears that there is an additional resistance which is independent of the agitation of the liquid.

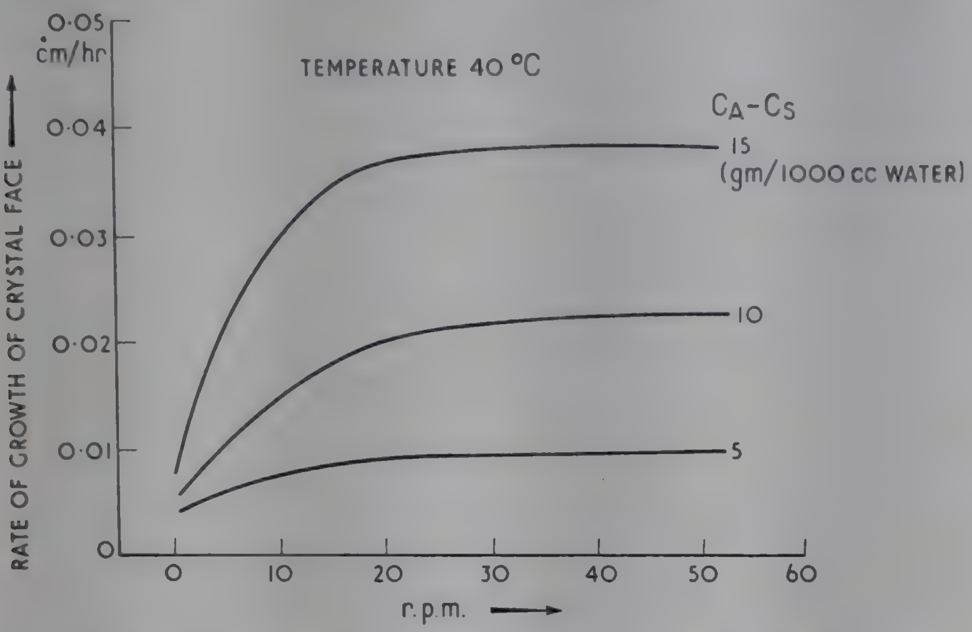

Fig. 22.5. Effect of speed of rotation on rate of growth of crystal of sodium thiosulphate

The rate of crystallisation is a function of the degree of supersaturation. For crystallisation from a melt, the process is dependent on the rate of transfer of heat between the crystal face and the surroundings. If $T_M$ is the melting point and $T_L$ is the temperature of the liquid, the apparent supercooling is $T_M - T_L$. Because of the conversion of latent heat into sensible heat as the material solidifies, the temperature at the interface rises to $T_i$ and the true supercooling is then only $T_M - T_i$ (Fig. 22.6). The temperature at the interface must clearly be greater than that of the bulk of the liquid since otherwise there would be no transfer of heat. The higher the heat transfer coefficient, the smaller is the required temperature difference $T_i - T_L$, and therefore a high rate of agitation will cause $T_i$ to approach $T_L$. Although there does not appear to be any regular relationship between the rate of growth of a single face of a crystal and the supercooling, there is a general tendency for a rise in the growth rate as the temperature difference is increased.

For crystallisation from a solution, very much less supercooling is possible —about 2 or 3 degrees as compared with 50 or 100 degrees for melts—and the process involves mass transfer rather than heat transfer, as already indicated. In this case the two processes of nucleation and growth cannot be so conveniently separated. Fig. 22.7 shows the concentration gradient in the neighbourhood of a crystal face. $C_S$ represents the concentration of a saturated solution and $C_A$ the concentration in the bulk of the liquid. The concentration falls from $C_A$ to $C_B$ through the liquid, and the concentration difference $C_B - C_S$ is required in order to overcome the resistance at the interface. Thus, whereas $C_A - C_S$ is the apparent supersaturation, the concentration difference which is responsible for mass transfer is only $C_A - C_B$. In this case the heat effects at the face are generally insignificant.

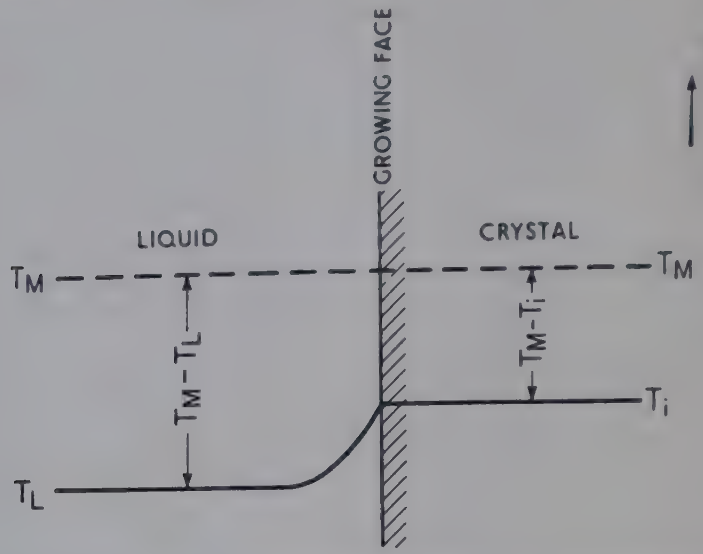

Fig. 22.6. Temperature distribution near surface of crystal

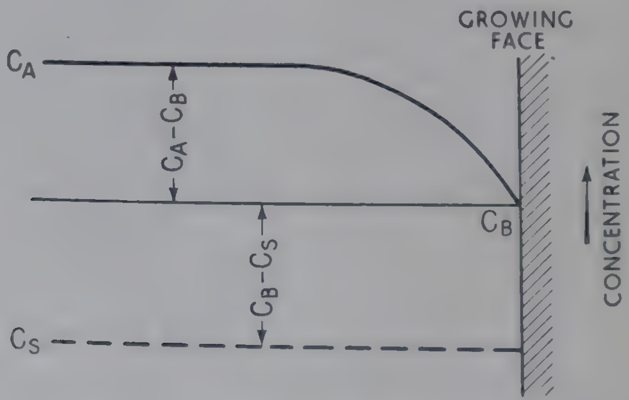

Fig. 22.7. Concentration near crystal.

The growth of a crystal face has been studied in detail by **Bunn**[14] who observed that the process was erratic in that there were irregular periods of rapid and of slow or zero rate. He also observed that the face grew by the building up of successive layers. Whereas the attachment of the first group of molecules to the surface is relatively difficult because of the weakness of the bonding, subsequent groups can add on quite readily as they are secured not only to the previous layer but also the material already deposited in the new layer. Thus, the crystal grows layer by layer even though, as reported by BUTLER(17), the concentration of solution at the surface varies over the face of the crystal (see Fig. 22.8). A recent theory of FRANK(15) suggests that deposition occurs at a dislocation in the structure of the crystal and that growth takes place in a spiral manner.

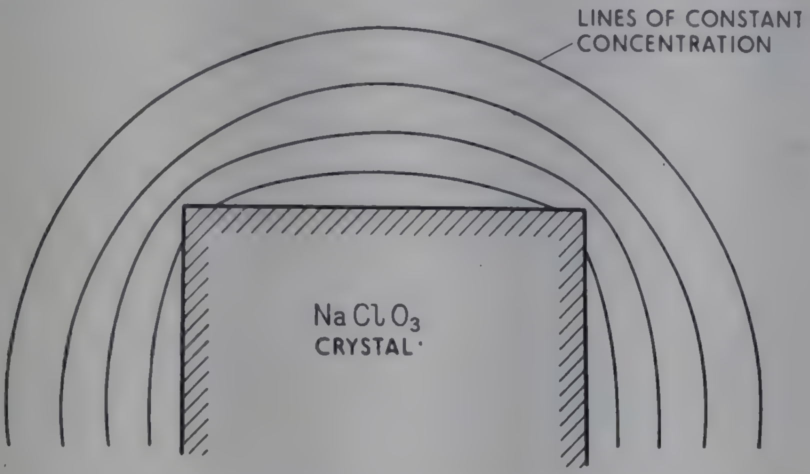

Fig. 22.8. Concentration of solution round crystal of $NaClO_3$

Although there is no relation for the instantaneous rate of growth of a crystal face, one can obtain a statistical relation over a period of time. NOYES and WHITNEY(4) have assumed that the diffusion of material to the crystal face is controlled almost entirely by the resistance of the laminar layer near the face. On this basis we can write

$$\frac{\mathrm{d}m}{\mathrm{d}t}=\frac{DA}{\delta_b}(C_A-C_B) \qquad \ldots.(22.2)$$

where $A$ is the area of the crystal surface,

$D$ is the diffusivity of the solute in the solution,

$\delta_b$ is the thickness of the laminar layer (it decreases as the degree of agitation is increased), and

$m$ is the mass of material deposited in time $t$.

BERTHOUD(6) assumed that the transfer at the interface was directly proportional to the supersaturation at the interface.

Thus
$$\frac{\mathrm{d}m}{\mathrm{d}t}=KA(C_B-C_S) \qquad \ldots.(22.3)$$

where $K$ is the rate constant for the surface process.

Eliminating $C_B$ between equations 22.2 and 22.3,

$$\frac{\mathrm{d}m}{\mathrm{d}t}=\frac{A}{\frac{1}{K}+\frac{\delta_b}{D}}(C_A-C_S) \qquad \ldots.(22.4)$$

BUTLER[17] has shown that the surface process is more complicated than is suggested by equation 22.3, and that

$$\frac{dm}{dt} \propto (C_A - C_S)^n \qquad \ldots.(22.5)$$

where $n$ is greater than unity.

Using the simplified equation (22.4), the linear rate of growth is given as

$$\frac{dl}{dt} = \frac{1}{\rho_s} \cdot \frac{1}{\dfrac{1}{K} + \dfrac{\delta_b}{D}} (C_A - C_S) \qquad \ldots.(22.6)$$

Then if $\frac{1}{K}$ and $\frac{\delta_b}{D}$ are both constant, we can write

$$\frac{dl}{dt} = k\Delta C \qquad \ldots.(22.6a)$$

Thus $\frac{dl}{dt}$ is independent of the actual size of the crystal. During growth, the shape of the crystal does not alter and therefore the large faces must grow less rapidly than the small ones. This is further evidence of a resistance to transfer at the crystal faces, because, if all the resistance were in the liquid film, each face would grow at the same rate. It is the variation of $K$ from face to face which allows the crystal to maintain its shape.

The ratio of the volume, to the cube of the linear dimension, must remain constant as the shape does not alter.

i.e. $$V = k'l^3, \text{ say} \qquad \ldots.(22.7)$$

and $$dV = 3k'l^2\, dl$$

Thus the rate of increase of volume is given by

$$\frac{dV}{dt} = 3k'kl^2\Delta C$$

$$\propto l^2\Delta C \qquad \ldots.(22.8)$$

From the above equations the rate of growth can be calculated approximately, but as in practice this may show considerable variation because, for instance, of the effect of small quantities of impurities, it is always necessary to check the results experimentally.

Because the linear rate of growth of a crystal is independent of its size, the change in the size distribution of a set of seed crystals can be calculated, on the assumption that the size range of these seeds is sufficiently small for the solubility of each crystal to be approximately the same. Thus if the seed crystals have dimensions in a particular direction of $l_1$, $l_2$, . . . etc., their volumes will be $k'l_1^3$, $k'l_2^3$ . . . etc. If during the period of growth of these crystals this dimension has increased by an amount $\Delta l$, the volumes of the crystals in the product will be $k'(l_1 + \Delta l)^3$, $k'(l_2 + \Delta l)^3$ . . . etc. Then if a total mass $M$ of solute is crystallised,

$$M = \rho_s k' \{\sum (l_1 + \Delta l)^3 - \sum l_1^3\} \qquad \ldots.(22.9)$$

This expression enables the change in the size distribution of the crystals to be calculated, after a known amount of solute has been removed from solution.

### Effect of Impurities on Crystal Formation

Although in many cases a soluble impurity will remain in the liquid phase so that pure crystals are produced, there are a number of instances where both the rate of nucleation and the rate of crystal growth may be affected. The effect is more usually a retardation and is often stated to be due to adsorption of the impurity on the surface of the nucleus or crystal. Thus materials with relatively large molecules, such as glue, tannin, dextrin or sodium hexametaphosphate (calgon), when added in small quantities to boiler feed water prevent nucleation and growth of calcium carbonate crystals and therefore reduce scaling. Further, addition of 0·1% HCl and 0·1% $PbCl_2$ will prevent the growth of sodium chloride crystals.

In certain cases the adsorption occurs preferentially on a particular face of the crystal with the result that the shape is modified. Thus sodium chloride, crystallised from solutions containing small quantities of urea, forms octohedral instead of the usual cubic crystals; these have a low packing density. Dyes are preferentially adsorbed in this way and their action is quite specific and not generally predictable.

Solid impurities in the fluid act as condensation nuclei but generally cause some dislocation in the crystal structure.

### Effect of Temperature on Solubility

Whereas increase in the temperature of the solution usually increases the solubility of the solute, there are instances where the temperature coefficient of solubility is negative, and sometimes it is zero. When the stable crystal-form changes as the temperature is altered, e.g. with hydrated salts, the curve is discontinuous; the coefficient may be positive over part of the range of temperature and negative over the remainder. A negative coefficient indicates a heat of reaction as the material passes into solution, which is greater than the heat absorbed due to the dissolution and dilution processes combined; anhydrous salts therefore tend to have negative coefficients. In Fig. 22.9 the solubility curves are given for a number of salts in water. Potassium chlorate has a large positive temperature coefficient and therefore is readily crystallised by cooling a saturated solution. Sodium chloride has a small coefficient and very little crystallisation occurs on cooling, and evaporation of solvent is therefore essential. The curves for sodium hydrogen phosphate and ferrous sulphate show a number of discontinuities which are attributable to changes in the stable form of the material. Thus crystallisation from a solution of ferrous sulphate at temperatures below about 50°C results in the formation of the hydrate $FeSO_4{\cdot}7H_2O$. On the other hand, crystallisation at higher temperatures will yield either $FeSO_4{\cdot}4H_2O$ or anhydrous $FeSO_4$. With careful regulation of temperature the material can be produced in the desired crystalline form. In many cases it is possible to obtain the hydrated salt only by use of comparatively low temperatures, and vacuum crystallisation is then frequently used.

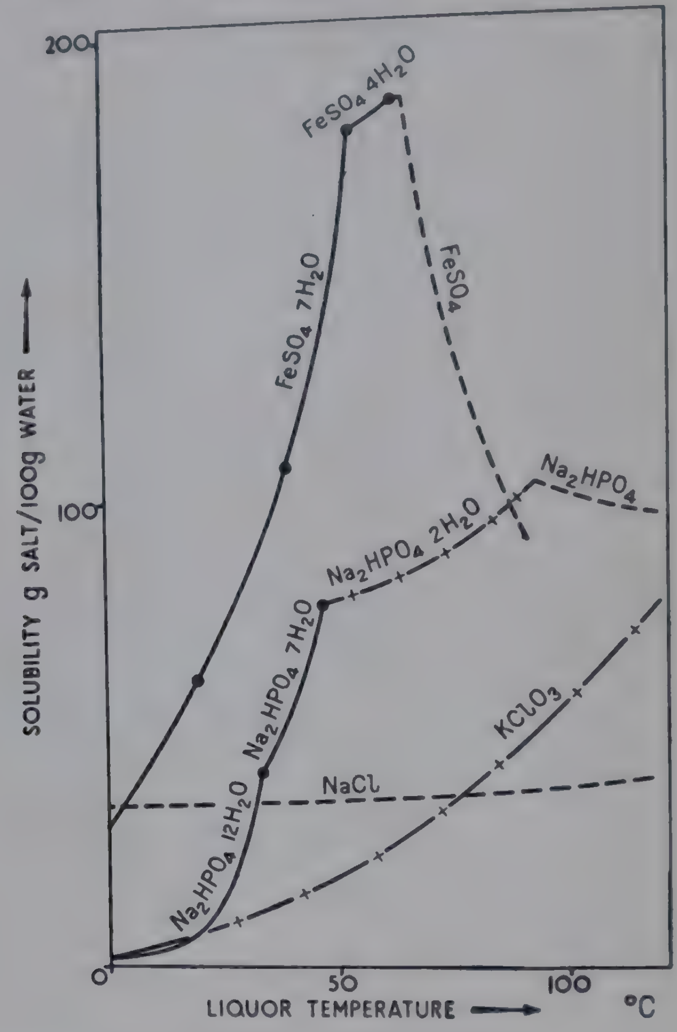

Fig. 22.9. Solubilities of salts in water.

## Fractional Crystallisation

It is often possible to separate a mixture of two soluble salts by fractional crystallisation and, in order to understand the process, it is necessary to consider the effect of additions of one salt on the solubility of the second when one ion is common to both salts. Fig. 22.10[2] illustrates the effect of additions of sodium chloride on the solubility of sodium nitrate at 120°C (line *AB*) and the effect of sodium nitrate on the solubility of sodium chloride (line *CB*). Line *AB* then represents mixtures which are saturated with respect to sodium nitrate but unsaturated with respect to sodium chloride, and *CB* represents the reverse condition. A mixture represented by *B* is saturated with respect to both salts and therefore represents the composition of the solution obtained by mixing an excess of solid with solvent. Curves *DE* and *FE* represent the corresponding conditions at 10°C, and some line joining *B* and *E* therefore represents the composition of solutions saturated with both components at various temperatures.

Separation of a mixed salt into its constituents is then effected, for instance, by forming a saturated solution at 120°, and allowing the solution to cool out of contact with the residual solid to 10°C. Since at this temperature a saturated solution contains more of sodium chloride and less of sodium nitrate, the latter will be deposited in pure form and the remaining solution will be unsaturated with respect to sodium chloride. The crystals can then be drained and washed, and a relatively pure product is obtained. The choice of conditions will affect the particle size and this in turn will affect the purity of the washed solids. If the crystals are relatively large they may tend to agglomerate so that mother liquor is occluded, whereas if they are too small drainage is difficult.

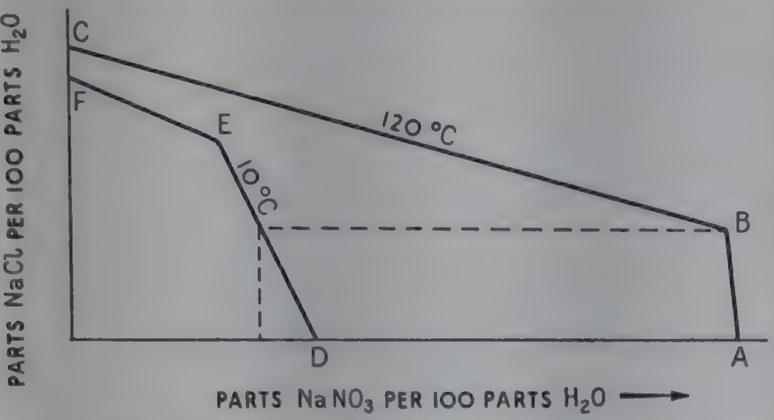

Fig. 22.10. Effect of sodium chloride on solubility of sodium nitrate

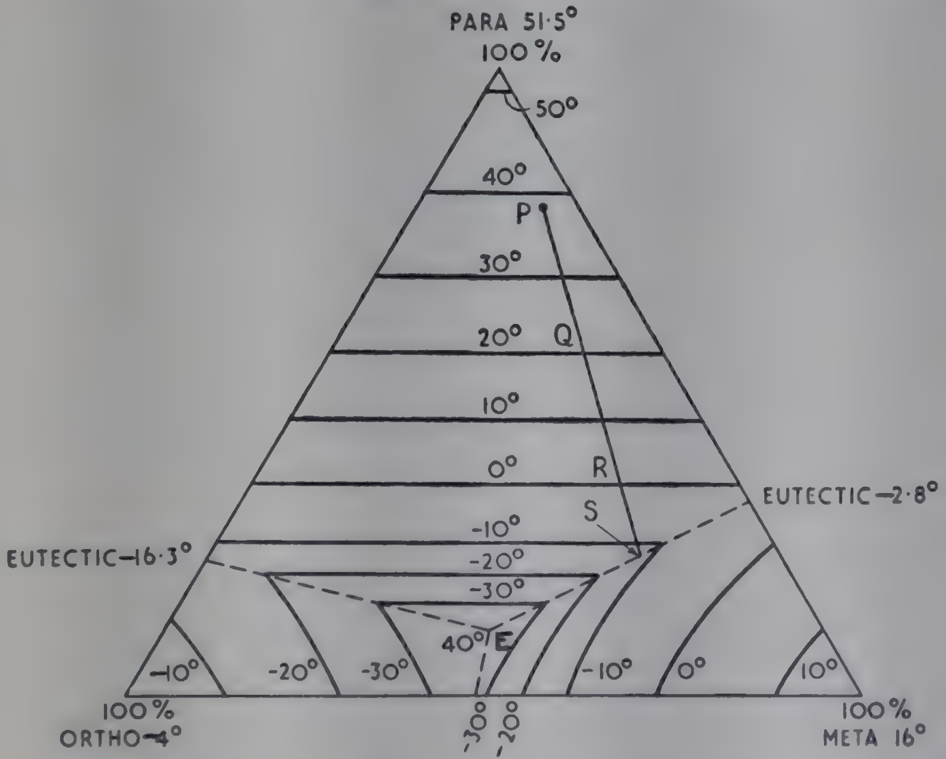

Fig. 22.11. Phase equilibrium diagram for ortho-, meta-, para-mononitrotoluenes

***Crystallisation of Mononitrotoluenes.***(3) As a further illustration of fractional crystallisation, we can take the separation of pure crystals of para-mononitrotoluene from a mixture of the three isomers, ortho, meta, and para. The equilibrium diagram for this system is shown in Fig. 22.11. If the hot liquor has a composition, 3% ortho, 8·5% meta and 88·5% para, then its composition is shown by point *P* on the diagram. For a mixture of this composition no crystals are deposited until the liquor is cooled to 46°C. On further cooling in the crystalliser, the composition of the remaining liquor is shown by the line *PQRS* for various temperatures indicated by the isothermals on the diagram. On cooling, only the para isomer is deposited and the ratio of ortho to meta remains constant till the point *S* is reached. Further cooling results in the meta being deposited along the line *SE*, till at point *E* all sets solid as a ternary eutectic. It is therefore undesirable to cool the mixture below point *S* (−15°C). If cooling water from a well is available, we should be able to cool the liquor down to 20°C and, as shown in Table 22.1, this will enable most of the para isomer to be removed.

Table 22.1. 100 lb of liquor cooled to 20°C.

| Component | Feed Liquor | | Mother Liquor | | Crystals Wt. lb |
|---|---|---|---|---|---|
| | % | Wt. lb | % | Wt. lb | |
| Ortho | 3·0 | 3·0 | 12·0 | 3·0 | — |
| Meta | 8·5 | 8·5 | 34·0 | 8·5 | — |
| Para | 88·5 | 88·5 | 54·0 | 13·5 | 75 |
| | 100·0 | 100·0 | 100·0 | 25·0 | 75 |

Cooling to lower temperatures gives an increasing weight of para crystals, as shown in Table 22.2. It is clear that with this system comparatively little is gained by cooling below 20°C, whereas the cost of cooling to lower temperatures is considerable. If, however, it was decided then to obtain the meta isomer as a pure material, cooling to a much lower temperature would be necessary in order to provide a liquor of sufficiently high concentration in meta to justify distillation. Thus, if we cool to −10°C, the percentage of meta in the mother liquor would be nearly 54 and it would be possible to obtain pure meta by distillation.

Table 22.2

100 lb of liquor of composition 3% o, 8·5% m, 88·5% p

| Temperature on cooling °C | Wt. of para as crystals lb |
|---|---|
| 46 | 0 |
| 30 | 66·7 |
| 20 | 75·0 |
| 10 | 79·6 |
| 0 | 82·3 |
| −10 | 84·2 |
| −18 | 84·8 |

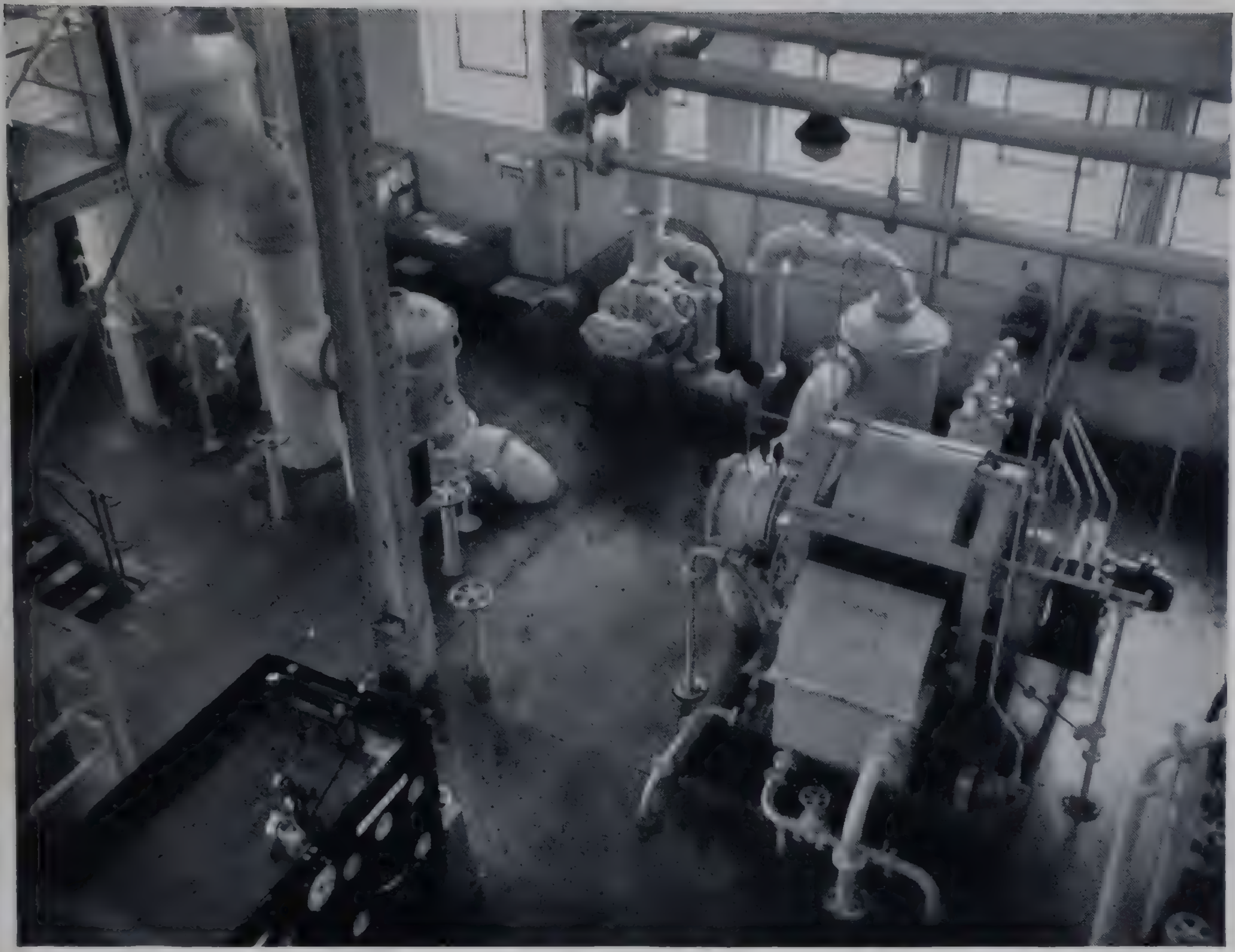

Fig. 22.12. Part of plant for recovery of sodium sulphate in viscose process. Crystallising evaporator on left. Rotary filter on right

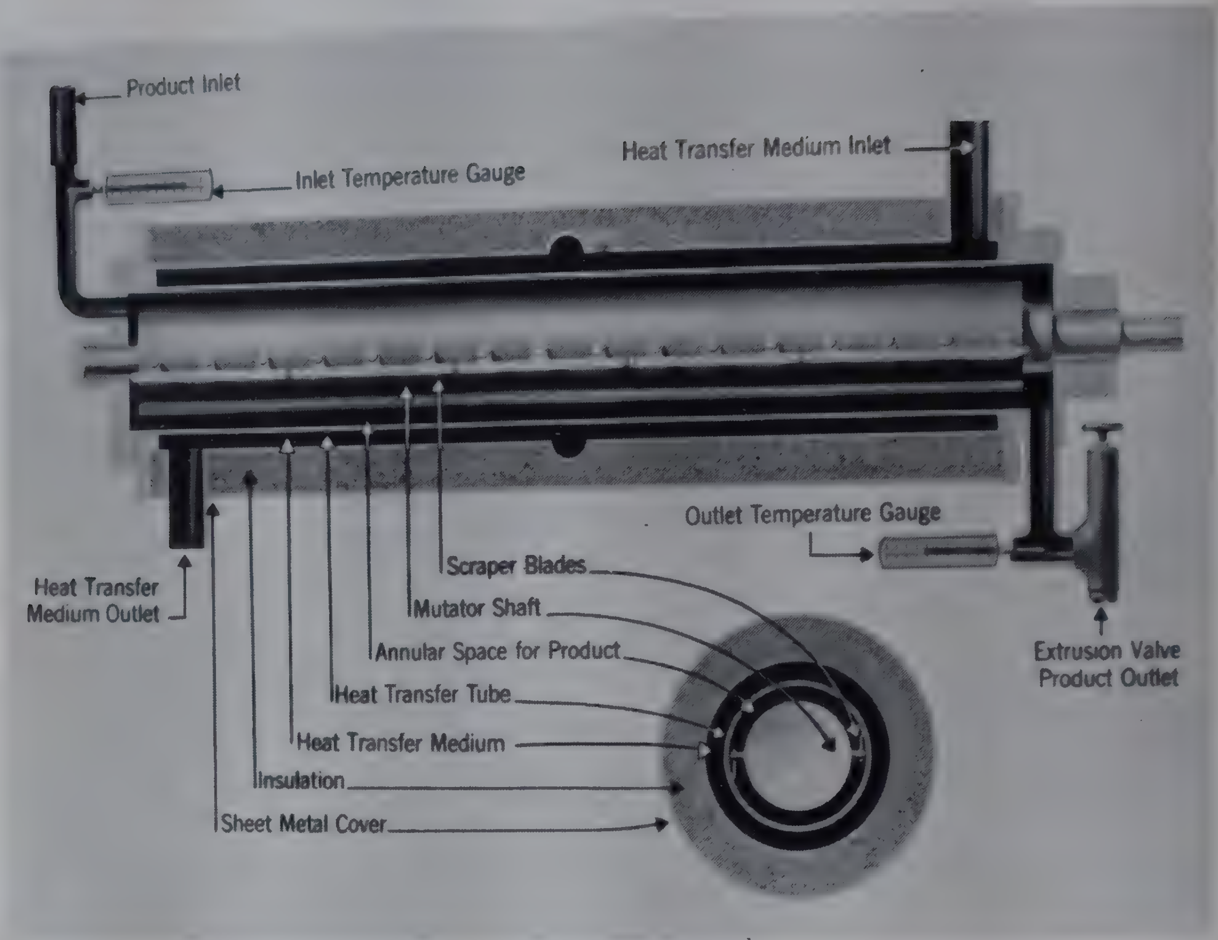
Product Inlet
Inlet Temperature Gauge
Heat Transfer Medium Inlet
Outlet Temperature Gauge
Scraper Blades
Mutator Shaft
Annular Space for Product
Heat Transfer Tube
Heat Transfer Medium
Insulation
Sheet Metal Cover
Heat Transfer Medium Outlet
Extrusion Valve Product Outlet

Fig. 22.13. The Votator apparatus

### Caking of Crystals

The tendency for crystalline materials to cake is attributable to a small amount of dissolution taking place at the surface of the crystals and subsequent re-evaporation of the solvent. The crystals can then become very tightly bonded together.

Because the vapour pressure of a saturated solution of a crystalline solid is less than that of pure water at the same temperature, condensation can occur on the surfaces of the crystals even though the relative humidity of the atmosphere is less than 100%. The solution so formed then penetrates into the pack of crystals by virtue of the capillary action of the small spaces between the crystals, and caking can occur if the absorbed moisture subsequently evaporates when the atmospheric humidity falls. Caking can also occur at a constant relative humidity, because the vapour pressure of a solution in a small capillary is lower than that in a large capillary, as a result of the Kelvin effect. As condensation takes place, the small particles are the first to dissolve and the average size of the capillaries is therefore increased, and the vapour pressure of the solution may increase sufficiently for evaporation to occur(16).

A crystalline material will cake more readily if the particle size is non-uniform, because the porosity is less in a bed of particles of mixed sizes and fine particles are more readily soluble. Thus, the propensity for caking can be reduced by forming crystals of relatively large and uniform sizes. Alternatively, small amounts of insoluble materials, such as calcium phosphate or talc, or of a water repellant agent, such as stearic acid or a stearate, may be added.

## CRYSTALLISERS

The more important types of plant used for producing crystals from solutions will now be described because this is the most common application in the chemical industry. Broadly, crystallisers may be classified according to whether they are batch or continuous in operation; continuous crystallisers can be divided into the linear and stirred types which will be referred to later. Crystallisers can also be classified according to the method by which supersaturation is achieved. In the evaporative crystalliser conditions are approximately isothermal, and supersaturation is achieved as a result of the removal of solvent. In the cooling crystalliser, supersaturation results from lowering of the temperature of the solution, and this can be effected either by means of exchange of sensible heat or by evaporative cooling; in the latter case, there is a small loss of solvent. Evaporative crystallisation must of course be used where the solubility shows little variation with temperature.

The main feature of the crystalliser is the method by which the size of the product is regulated, and this is almost entirely dependent on the control of the nucleation process. In general, slow cooling will result in the formation of relatively few and large crystals because spontaneous nucleation is then reduced, and rapid cooling will result in a high rate of nucleation and a small size of crystal. Crystallisers are generally simple in construction, the only moving parts being agitators and the scrapers which are used to keep the heat transfer surfaces free of solids.

## Batch Crystallisers

***Tank Crystallisers.*** The simplest and cheapest type of crystalliser consists of an open tank, which can be used either as an evaporative or as a cooling crystalliser. In the former case, it is generally heated by means of steam coils or from a jacket. In the latter case, the solvent is evaporated until the concentration has reached the required value, and cooling is then effected by transfer of sensible heat to the surroundings and evaporation at the free surface. The solution cools slowly and large crystals are therefore obtained but they are generally uneven because the nucleation is not controlled in any way. The tank is normally ridged to facilitate removal of the mother liquor, but even so drainage is usually incomplete. Labour costs are high for this type of plant. Salt for the fishing industry can be prepared using long shallow open pans heated by steam coils. As the solubility is almost independent of temperature, the crystals form in the liquid surface and are held there by surface tension forces until they exceed a certain weight, when they fall to the bottom and are removed by rakes. Hard saucer-shaped crystals are obtained by this method.

The capacity of the simple tank crystalliser can be increased, and the uniformity of the product improved, by using an agitator and a series of cooling coils. The main disadvantage is that crystals form on the surface of the coils and seriously impede the heat transfer, but it is sometimes possible to brush the surfaces free of crystals. Stainless steel construction is often used because particles do not adhere so firmly as to other materials.

***Evaporators.*** Salting evaporators are frequently used for crystallisation and these have already been referred to in Chapter 21. They are generally calandria type evaporators with relatively short wide tubes and a large downcomer, so designed that the crystals do not cause obstruction. The solution is concentrated and then withdrawn, either using a salt box or through a barometric leg. The crystals are separated from the mother liquor which is then returned to the evaporator together with the feed, and evaporation is continued until the concentration of impurities in the solution becomes excessive. When used in this way evaporators may be operated either at atmospheric or at reduced pressure; reduced pressures are essential for the formation of many hydrated crystals. Fig. 22.12 shows part of a plant using a crystallising evaporator.

***The Use of Vacuum.*** Crystallisers employing evaporative cooling often operate under vacuum, and the hot feed is introduced into the body which generally consists of a tall vertical cylinder; a low pressure is maintained by means of a steam jet ejector. Flash evaporation of the solution takes place and produces rapid cooling accompanied by a small increase in concentration. The suspension of crystals is then pumped out, either continuously or in batches. Because these plants operate at very low pressures, cooling water is not generally available at a low enough temperature to condense the vapour, and the vapour is therefore compressed before it enters the condenser; the size of the condenser is thereby considerably reduced in addition. In many cases the solution is agitated or continuously circulated by an external pump in order to prevent stratification.

Vacuum crystallisation is frequently used in the sugar industry where a concentrated sugar solution is fed to an evaporator operating under reduced pressure. Concentration is continued until nucleation commences, when the solution is suddenly cooled by flash evaporation by increasing the vacuum. When nucleation is complete, fresh solution is added and evaporation is continued so as to produce crystals of the required size.

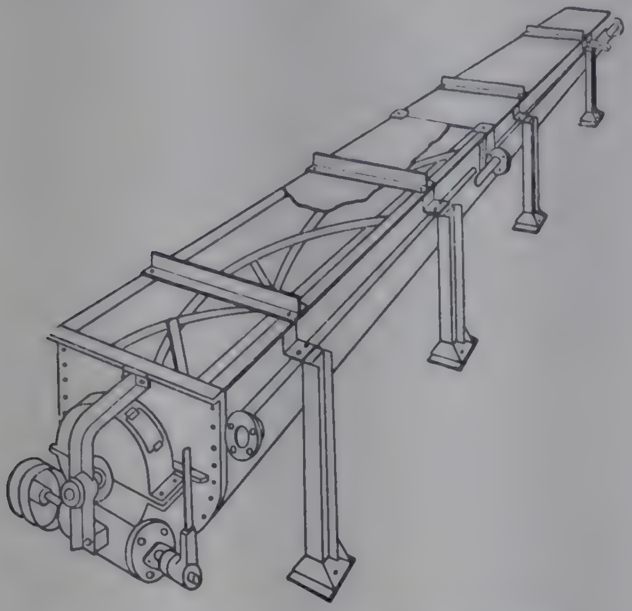

Fig. 22.14. Swenson-Walker crystalliser

## Continuous Crystallisers

There are two main classes of continuous crystallisers; the linear type in which the solution flows along a trough or pipe with very little longitudinal mixing, and the stirred type in which uniform conditions are maintained.

***Swenson-Walker Crystalliser.*** The Swenson-Walker crystalliser (Fig. 22.14) is of the linear type and consists of a long open trough about 2 feet across and divided into a number of sections, each of which can be independently cooled with water in an external jacket, so that it is possible to control the rate of cooling throughout. A spiral scraper keeps the cooling surfaces free of crystals. Sometimes small seed crystals are added to form the nuclei, but more often spontaneous nucleation is effected in the first section by suitable adjustment of the temperature. Slow cooling is obtained in subsequent sections, and the solution may be cooled below atmospheric temperature by using refrigerated brines in the jacket. This crystalliser is suitable only where supersaturation can be effected by cooling alone. It gives fairly uniform crystals but some breakage is caused by the scraper.

***Wulff-Bock Crystalliser.*** The Wulff-Bock Crystalliser (Fig. 22.15) has similar characteristics to the Swenson-Walker but relies on air cooling and gives more uniform crystals. It consists of a trough which is slowly rocked from side to side; transverse baffles, fitted alternately to opposite sides, prevent surging. Because there are no moving parts, it is easily fabricated from corrosion-resistant materials, and sometimes it is rubber lined.

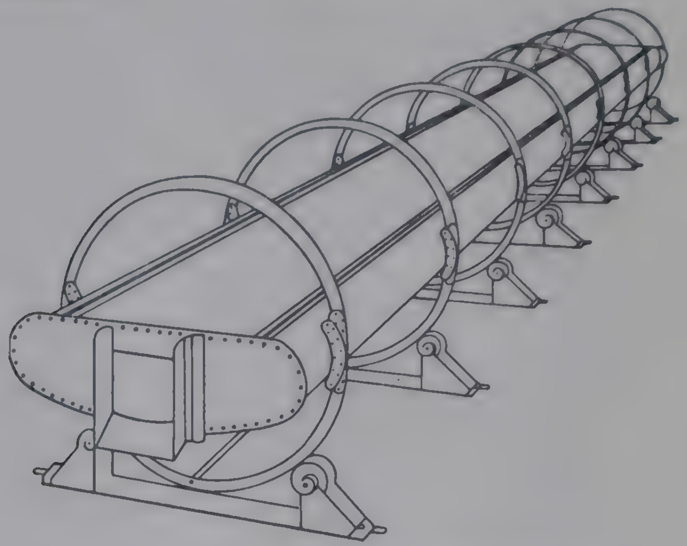

Fig. 22.15. Wulff-Bock crystalliser

***The Double-Pipe Cooler Crystalliser or Votator Apparatus.*** The Votator Apparatus is a linear type cooler crystalliser in which the heat transfer medium flows through an annular jacket and the liquid undergoing treatment passes through the narrow space between the heat transfer surface and the shaft of the mutator which carries scraper blades (Fig. 22.13). By virtue of the high degree of turbulence and the continuous scraping of the surface, very high heat transfer coefficients are obtained. The Votator Apparatus is very suitable for highly viscous or heat-sensitive liquids. It is extensively used for crystallising paraffin wax in the petroleum industry and in the plasticizing of margarine and cooking fats, and has many other applications.

***The Krystal or Oslo Crystalliser.*** The Oslo crystalliser was first developed in Norway(11), and is now extensively used where large quantities of crystals of controlled size are required. The plant is made in a number of different forms, but in each the basic principle is to pass a supersaturated solution upwards through a bed of crystals which are maintained in a fluidised state. A uniform temperature is thereby attained, and the crystals segregate in the bed with the small ones at the top and the large ones at the bottom. A typical form of crystalliser is shown in Fig. 22.16 from which it is seen that mother liquor is withdrawn near the feed point $E$ of the crystalliser by a circulating pump, and is passed through the cooler $H$ where it becomes supersaturated, and then is fed back to the bottom of the crystalliser through the central pipe $B$. The feed forms only a small proportion of the total liquid circulating, and nucleation does not generally take place in the cooler. Some nuclei form spontaneously in the crystal bed and some form as a result of breakage of the crystals. These nuclei circulate with the mother liquor until they have grown sufficiently large to be retained in the fluidised bed. The final product is removed entirely from the bottom of the crystalliser through a valve

*M*, and a uniform product is thereby obtained because the crystals are not discharged until they have grown to the required size. A small amount of fines is often formed as a result of mechanical attrition, however. In some cases channelling of the bed occurs, but this can be prevented by introducing the solution through a perforated plate or gauze.

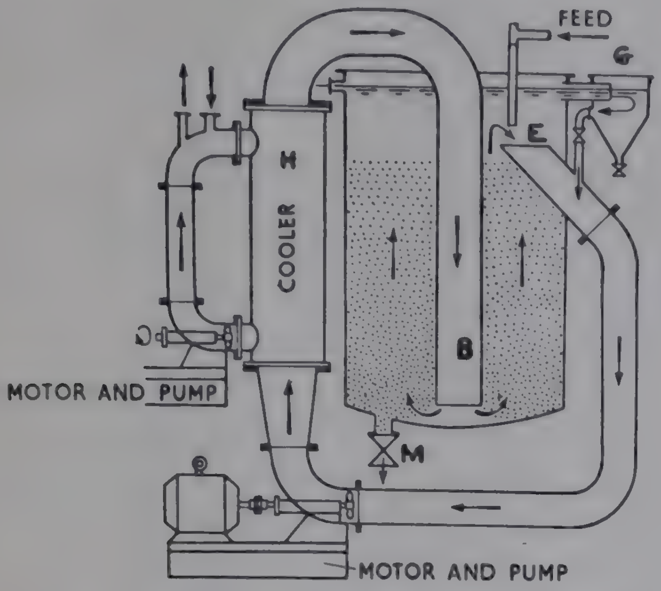
FEED
G
E
H
COOLER
B
M
MOTOR AND PUMP
MOTOR AND PUMP

Fig. 22.16. Oslo cooler crystalliser

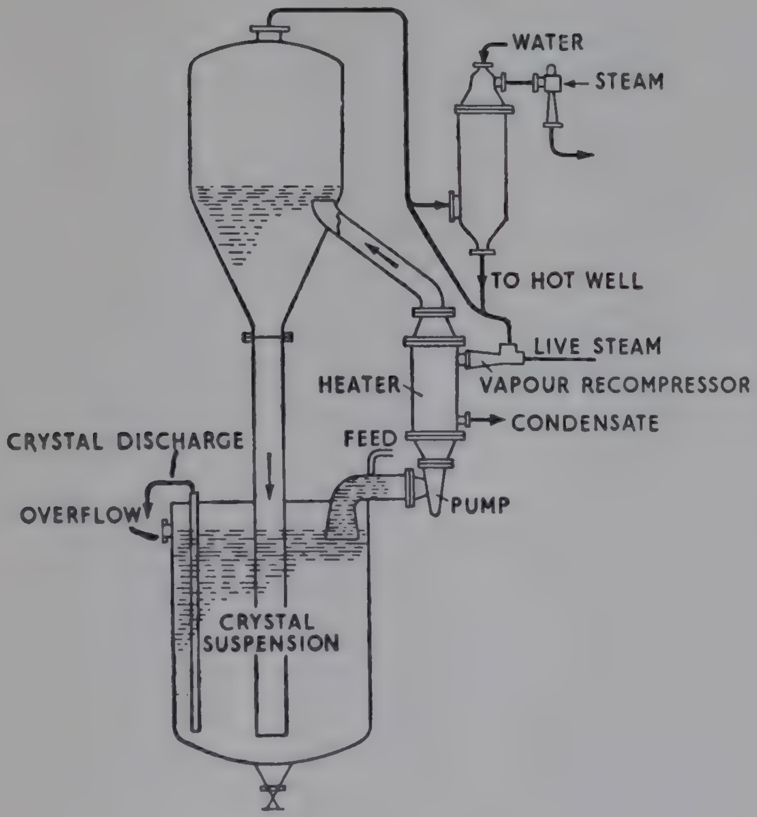
WATER
STEAM
TO HOT WELL
LIVE STEAM
VAPOUR RECOMPRESSOR
HEATER
CONDENSATE
CRYSTAL DISCHARGE
FEED
OVERFLOW
PUMP
CRYSTAL SUSPENSION

Fig. 22.17. Oslo evaporative crystalliser

In the crystalliser just described there is no provision for evaporation, and therefore modifications are necessary for salts whose solubilities have small temperature coefficients. In the plant shown in Fig. 22.17, the solution is first passed through a heater and then to a flash evaporator before being returned to the crystalliser. This type can be operated under reduced pressures for the production of hydrated crystals, and multiple-effect evaporation is sometimes used.

Control of crystal formation is generally easier in the stirred than in the linear type of crystalliser. In the Oslo cooling crystalliser, the following are the main control variables.

(*a*) The feed rate.
(*b*) The feed temperature.
(*c*) The heat removed in the cooler.
(*d*) The circulation rate.
(*e*) The rate of removal of nuclei.

The effect of altering any one of these quantities may be quite complex, but the general picture is as follows.

(*a*) Increasing the feed rate causes the material to remain in the crystalliser for a shorter time. The capacity is increased but the crystals will be smaller, and the concentration of the discarded mother liquor will be greater.

(*b*) At high feed temperatures, it is possible to obtain a greater throughput, provided the additional heat is removed in the cooler, because a more concentrated feed can be used.

(*c*) If the heat removed in the cooler is increased, a greater yield is obtained. The crystals are smaller because there is a higher degree of supersaturation.

(*d*) The liquid must remain in contact with the crystals sufficiently long for it to lose its supersaturation. If the rate is too high, supersaturated liquid will be fed to the pump. If it is too low, all the supersaturation will be lost before the solution has reached the top of the crystal bed, and the upper portions of the bed will be ineffective.

(*e*) Nuclei may be withdrawn from the mother liquor before it enters the cooler, by means of a decanter (*G* in Fig. 22.16). The solution obtained can then be reheated so that the nuclei dissolve before it is returned to the crystalliser. The fewer the nuclei present, the larger will be the final crystals.

A typical Oslo crystalliser for the production of $MgSO_4 \cdot 7H_2O$ is fed with saturated solution at 120°C. The temperature of the plant is 40°C. With a feed of 400 gallons per hour and a circulation of 10,000 gallons per hour, the circulating liquid becomes heated to 43°C.

Oslo crystallisers are made in very large sizes, with bodies up to 15 feet in diameter and 20 feet high. The internal surfaces must be carefully smoothed so that there is no possibility of nuclei becoming lodged.

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## LIST OF SYMBOLS USED IN CHAPTER 22

| | | |
|---|---|---|
| $A$ | Area of crystal face | $L^2$ |
| $C_A$ | Concentration in bulk of solution | $ML^{-3}$ |
| $C_B$ | Concentration at interface | $ML^{-3}$ |
| $C_S$ | Concentration of saturated solution | $ML^{-3}$ |
| $D$ | Diffusivity of solute in solution | $L^2T^{-1}$ |
| $K$ | Rate constant for surface process | $LT^{-1}$ |
| $k$ | Coefficient relating rate of growth and supersaturation | $M^{-1}L^4T^{-1}$ |
| $k'$ | Volume coefficient | — |
| $l$ | Linear dimension of crystal | L |
| $M$ | Mass of solute crystallised | M |
| $m$ | Mass of solute deposited in time $t$ | M |
| $n$ | Index | — |
| $P_r$ | Vapour pressure at convex surface of radius $r$ | $ML^{-1}T^{-2}$ |
| $P_s$ | Vapour pressure at plane surface | $ML^{-1}T^{-2}$ |
| $r$ | Radius of droplet | L |
| $T_i$ | Temperature of crystal face | $\theta$ |
| $T_L$ | Temperature of bulk of liquid | $\theta$ |
| $T_M$ | Melting point | $\theta$ |
| $t$ | Time | T |
| $V$ | Volume of crystal | $L^3$ |
| $\delta_b$ | Thickness of laminar layer | L |
| $\rho_L$ | Density of liquid | $ML^{-3}$ |
| $\rho_S$ | Density of crystal | $ML^{-3}$ |
| $\rho_V$ | Density of vapour | $ML^{-3}$ |
| $\tau$ | Interfacial tension | $MT^{-2}$ |

## CHAPTER 23

# *Drying*

THE drying of materials is frequently the last operation in a manufacturing process, and is carried out immediately prior to packaging or dispatch. By drying, we usually refer to the final removal of water, and the operation often follows evaporation, filtration, or crystallisation. In some cases, drying is an essential part of the manufacturing process, as for instance in paper making or the seasoning of timber, but, in the majority of processing industries, drying is carried out for one or more of the following reasons:

(1) To reduce the cost of transport.
(2) To make a material more suitable for handling; e.g., soap powders, dyestuffs, fertilisers.
(3) To provide definite properties; e.g., to maintain the free flowing nature of salt.
(4) To avoid the presence of moisture which may lead to corrosion; e.g., the drying of coal gas, or the drying of benzene prior to chlorination.

With a crystalline product, it is necessary to ensure that the crystals are not damaged, and, with pharmaceutical products, care must be taken to avoid contamination. Shrinkage (as with paper), cracking (as with wood) or loss of flavour (as with fruit) must also be prevented. Leaving out of consideration the partial drying of a material by squeezing in a press or the removal of water by adsorption, almost all drying processes involve the removal of water by vaporisation, and thus require the addition of heat. In assessing the efficiency of a drying process, the effective utilisation of the heat will be a major criterion.

## SOME GENERAL PRINCIPLES

The moisture content of a material is usually expressed as a percentage of the weight of the dry material. If a material is exposed to air at any definite temperature and humidity, the material will either lose or gain water until an equilibrium condition is established. The equilibrium moisture content varies widely with the moisture content and the temperature of the air, as shown in Fig. 23.1. A non-porous insoluble solid, such as sand or China clay, will have an equilibrium moisture content approaching zero for all humidities and temperatures, but many organic materials, such as wood, textiles and leather, show wide variations of equilibrium moisture content, as indicated. The moisture may be present in two forms:

*Bound Moisture.* This is water retained in such a way that it exerts a vapour pressure less than that of free water at the same temperature. Such water may be retained in small capillaries, adsorbed on surfaces, or in solution in cell walls.

Defn

*Free Moisture.* This is the water that is in excess of the equilibrium moisture content.

The water removed by vaporisation is generally carried away by air or hot gas, and the ability of these gases to pick up the water will be determined by their temperature and humidity. In designing dryers using air, it is necessary to have available the properties of the air-water system in a convenient form. These properties have been discussed in detail in Volume 1, Chapter 10, where the development of the humidity chart has been described. Some of the more important factors are briefly described below for the *air-water system.*

*Humidity $\mathscr{H}$.* Mass of water carried per unit mass of dry air.

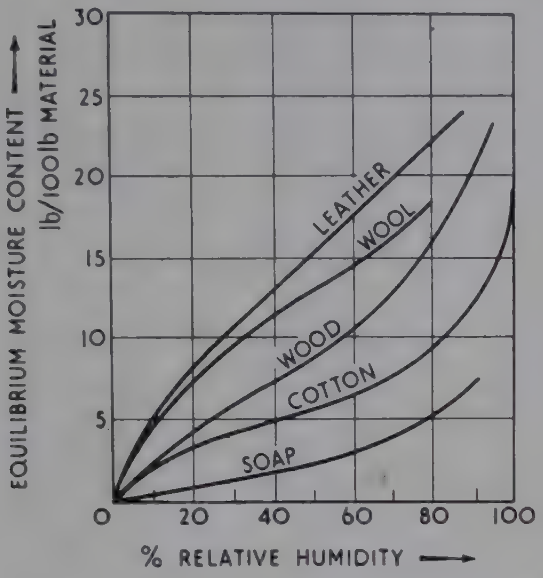


Fig. 23.1. Equilibrium moisture content of solid as a function of relative humidity (20°)

Since
$$\frac{\text{mols of water vapour}}{\text{mols of dry air}} = \frac{P_w}{P - P_w},$$

$$\mathscr{H} = \frac{18P_w}{29(P - P_w)}$$

where $P_w$ is the partial pressure of water vapour, and $P$ is the total pressure.

*Humidity of saturated air $\mathscr{H}_0$.* Humidity of air when saturated with water vapour. The air is in equilibrium with water at the given temperature and pressure.

*Percentage humidity.*
$$\frac{\text{Humidity of air}}{\text{Humidity of saturated air}} \times 100$$

$$= \frac{\mathscr{H} \times 100}{\mathscr{H}_0}$$

*Humid heat.* Heat required to raise unit mass of dry air and associated vapour through 1° at constant pressure, $= 0{\cdot}24 + 0{\cdot}45\mathscr{H}$.

*Percentage relative humidity.*

$$\frac{\text{Partial pressure of water vapour in air}}{\text{Vapour pressure of water at the same temperature}} \times 100$$

*Humid volume.* Volume of unit mass of dry air and associated vapour. At atmospheric pressure, this is equal to

$$\frac{359}{29}\,\frac{T}{273} + \frac{359\mathscr{H}}{18}\,\frac{T}{273}\ \text{ft}^3/\text{lb, where } T \text{ is in degrees Kelvin,}$$

or

$$\frac{359}{29}\,\frac{T}{492} + \frac{359\mathscr{H}}{18}\,\frac{T}{492}\ \text{ft}^3/\text{lb, where } T \text{ is in degrees Rankine.}$$

*Saturated volume.* Volume of unit mass of dry air, together with the water vapour required to saturate it.

*Dew point.* Temperature at which condensation will first occur when air is cooled.

*Wet bulb temperature.* If a stream of air is passed rapidly over the surface of water, vaporisation occurs, provided the temperature of the water is above the dew point of the air. The temperature of the water falls and heat flows from the air to the water. If the surface is sufficiently small for the condition of the air to change inappreciably and if the velocity is in excess of about 15 ft/sec, at equilibrium the water reaches the wet bulb temperature $\theta_w$.

Rate of heat transfer from gas to liquid

$$= hA(\theta - \theta_w) \qquad \ldots.(23.1)$$

Mass rate of vaporisation

$$= \frac{h_D A M_w}{RT}(P_{w0} - P_w) \qquad \text{(lb/hr, say)}$$

$$= \frac{h_D A M_A}{\mathbf{R}T}(P - P_w)_{\text{mean}}(\mathscr{H}_w - \mathscr{H})$$

$$= h_D A \rho_A(\mathscr{H}_w - \mathscr{H}) \qquad \ldots.(23.2)$$

Heat transfer required to effect vaporisation at this rate

$$= h_D A \rho_A(\mathscr{H}_w - \mathscr{H}) \,.\, \lambda \qquad \ldots.(23.3)$$

At equilibrium, the rates of heat transfer given by equations 23.1 and 23.3 must be equal.

Hence

$$\mathscr{H} - \mathscr{H}_w = -\frac{h}{h_D \rho_A \lambda}(\theta - \theta_w) \qquad \ldots.(23.4)$$

and the wet bulb temperature $\theta_w$ depends only on the temperature and humidity of the drying air.

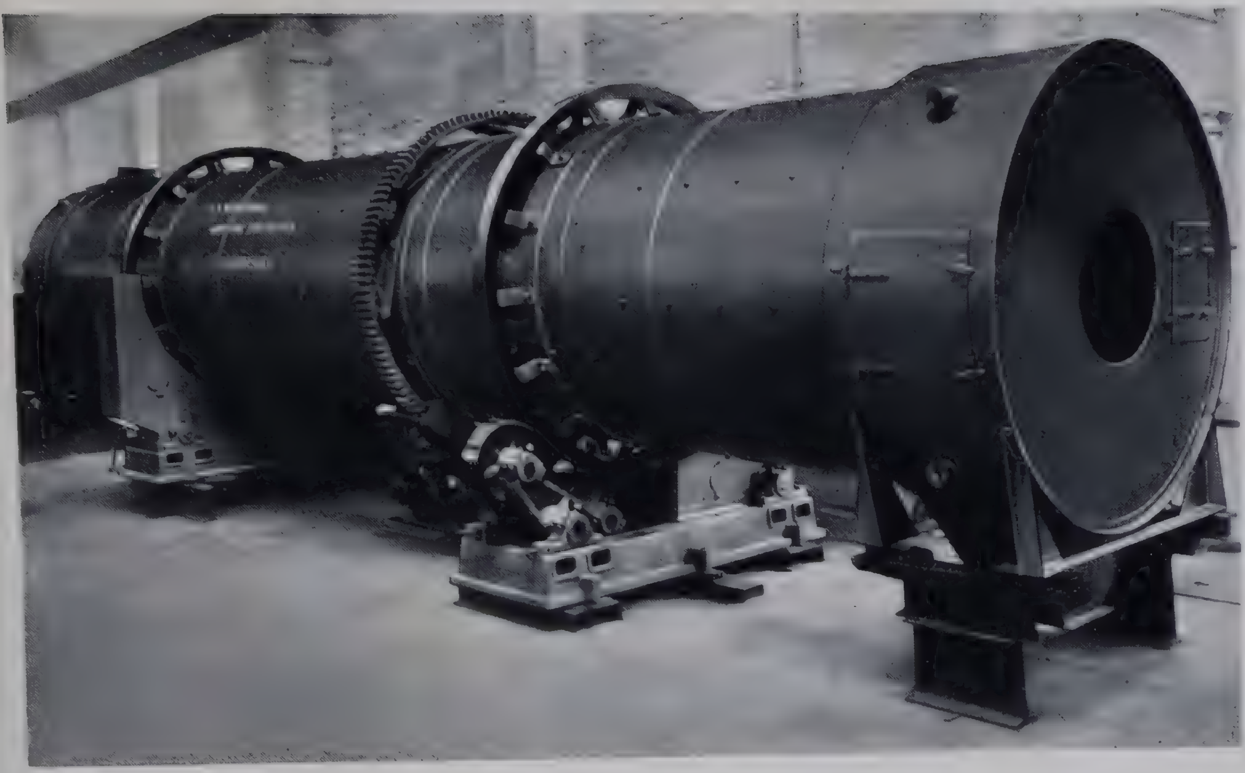


Fig. 23.7. Rotary dryer

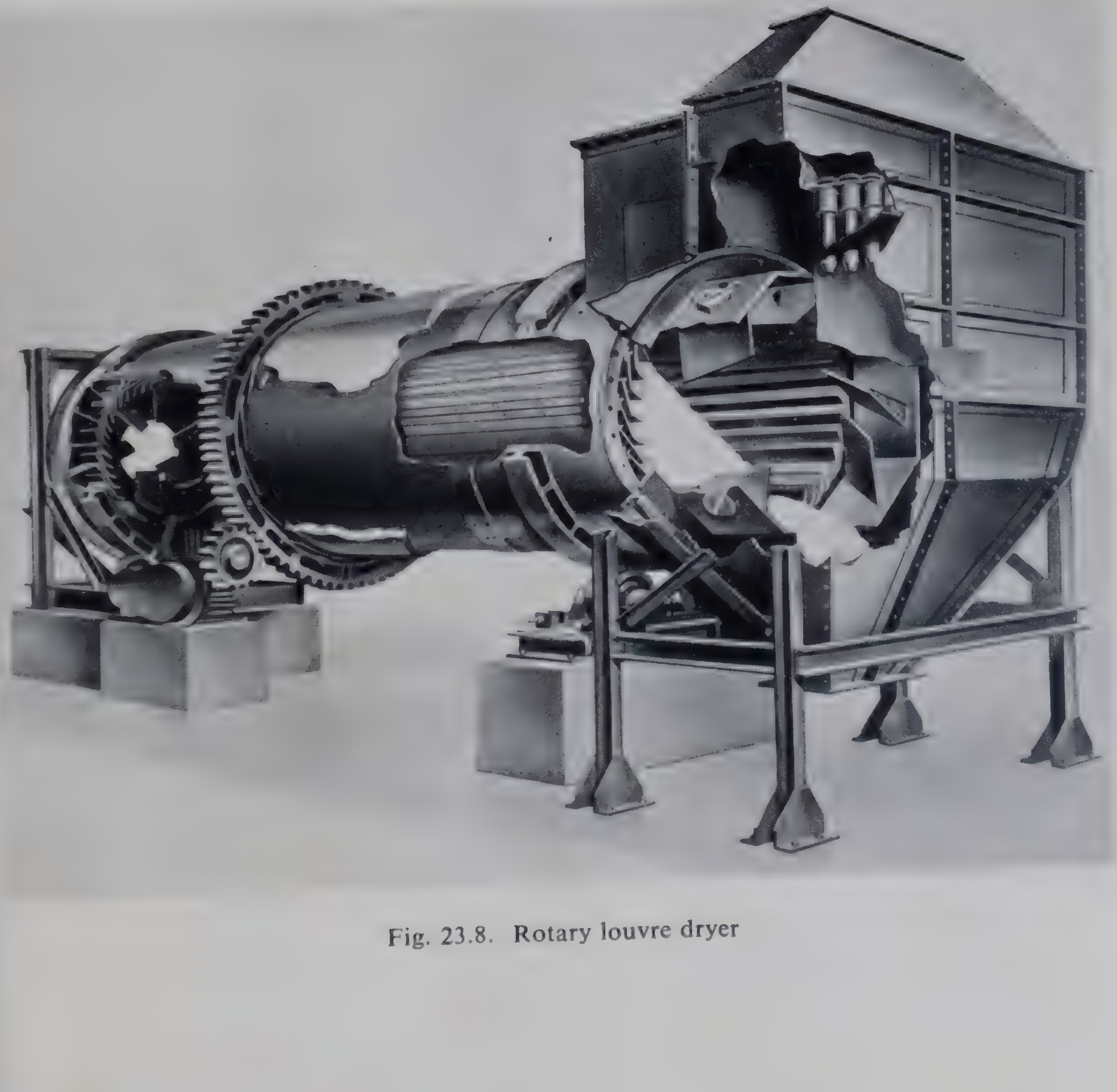


Fig. 23.8. Rotary louvre dryer

water from a single nozzle thins to a thickness of less than 5 microns, and instability is reached when the depth of any disturbance is of this order. To open the sheet against the forces of surface tension, a minimum sheet velocity must be produced by the pressure. Increasing the pressure opens the sheet and lengthens it to an optimum value.

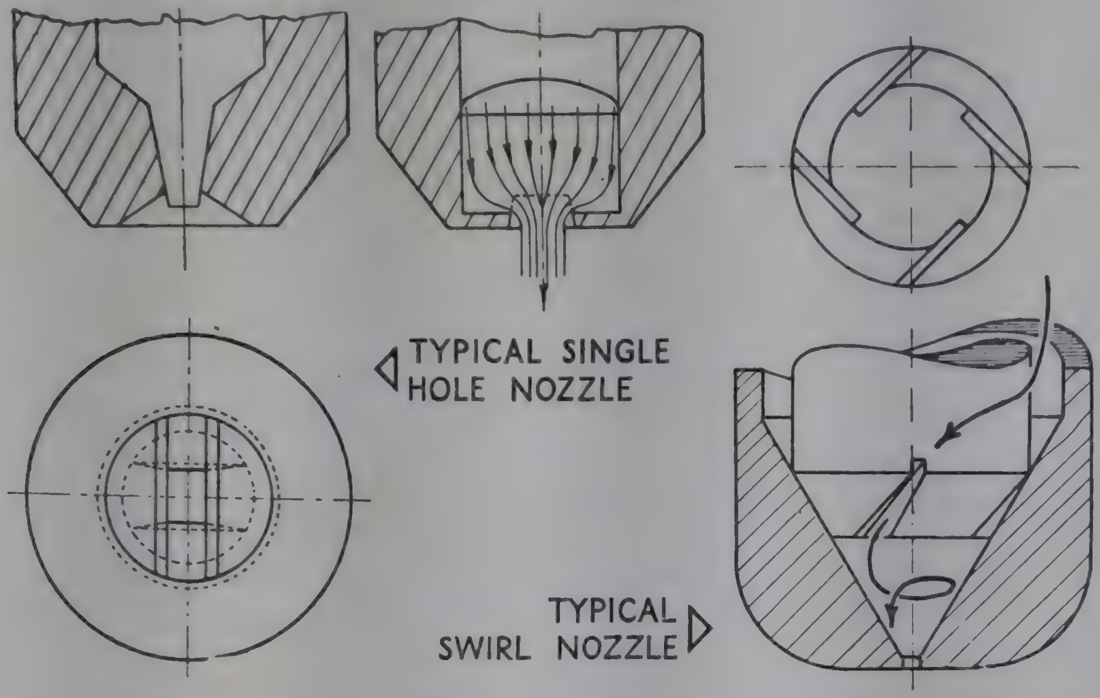


Fig. 23.13. Pressure nozzles

The mechanism of disintegration is the break up of a liquid thread, and this is shown in Fig. 23.18. Because of surface tension, the liquid rims are continually contracting giving "rim" disintegration. The threads are pulled out from the straight rims during contraction, and give rise to larger drops than from the edge of the sheet. In "perforated sheet" disintegration, the free edge disintegrates through a network of threads. Point disturbances on the sheet puncture it when they reach a thin enough region, and the resulting holes expand and coalesce and the threads become unstable and disintegrate. With "wavy" disintegration, the major wave disturbances caused by the surrounding atmosphere disintegrate the sheet by tearing surfaces away. It is the aim to give ordered disintegration as this gives a narrow range of drop sizes; this is effected by controlling the region of disintegration.

***Spinning Disc Atomisers.*** Experiments with discs up to 10·4 cm diameter and with speeds up to 2,500 r.p.m. have shown that the mean drop diameter can be obtained from the relation

$$d_m = \frac{0{\cdot}6}{N}\sqrt{\frac{\sigma}{D.\rho}} \qquad \ldots.(23.10)$$

where $d_m$ is the main drop diameter,
$N$ is the number of revolutions per unit time,
$\sigma$ is the surface tension,
$\rho$ is the liquid density, and
$D$ is the disc diameter.

With the spinning disc atomiser, the sheet thins out along the disc until its thickness at the rim is suitable for immediate drop formation. If the flow rate is increased, the sheet expands beyond the rim and break up occurs from an irregular free edge by "perforated" or "wavy sheet" disintegration. At the low flow rate the drops are of uniform size, but with the higher rates there is a wide range in size.

***Industrial Equipment.*** One common form of industrial rotary atomiser consists of a rapidly rotating inverted bowl fed with an annular stream directed on to a plate fixed near the apex of the bowl. The feed is thrown on to the inside of the bowl and leaves as a spray from the rim. The large dryers use discs of about 12 cm diameter, at speeds of 30,000 to 50,000 r.p.m.

In the food industry, where indirectly heated air is used, steam heating gives a maximum air temperature of about 170°C, but higher air temperatures (250–310°C) are obtained with gas or oil fired heaters. GROSE and DUFFIELD(23) have given the following figures for the cost of removing water by spray drying, and the high cost when using steam for air heating is clearly indicated.

TABLE 23.2. *Operating costs for a spray dryer evaporating 1 ton per hour of water*

Basis: Air outlet temperature 80°C.
Steam £1 per ton
Fuel Oil £15 per ton
Power 1d. per h.p. hour

| *Air heated* | *Air inlet Temp °C* | *Steam tons/hr* | *Oil tons/hr* | *Power b.h.p.* | *Cost per 100 hrs £* | | |
|---|---|---|---|---|---|---|---|
| | | | | | *Fuel* | *Power* | *Total* |
| Indirectly | 180 | 2·55 | — | 95 | 255 | 38 | 293 |
| Direct combustion | 250 | — | 0·12 | 74 | 180 | 30 | 210 |
| Direct combustion | 330 | — | 0·11 | 63 | 165 | 25 | 190 |

These figures indicated the relatively high cost of the process, but for many products it is undoubtedly the most satisfactory.

A semi-industrial scale spray dryer is illustrated in Fig. 23.19 (facing p. 849).

**Pneumatic Dryer**

In the pneumatic dryer illustrated in Fig. 23.14, the material is kept in a state of fine division, so that the surface per unit volume is high and a high rate of heat transfer is obtained. The solid is introduced into the dryer by some form of mechanical feeder, such as a rotating star wheel, or by an extrusion machine arranged with a high speed guillotine to give short lengths of material, say ¼ in. to ½ in. Hot gases from a furnace, or more frequently from an oil burner. are passed into the bottom of the dryer, and these pick up the particles and carry them up the column. The stream of particles leaves the dryer through a cyclone separator and the hot gases pass out of the system. In some instances, final collection of the fine particles is in a series of bag filters. In this unit the time of contact of particles with the gases is small and they do not approach the temperature of the hot gas stream.

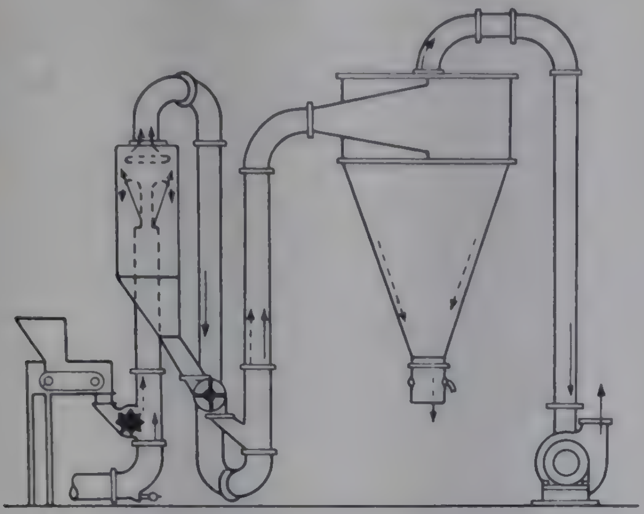


Fig. 23.14. Pneumatic dryer

### Turbo-Shelf Dryers

The handling of sticky material presents many difficulties which have not been entirely solved. One machine that is useful for this type of material is the turbo dryer, made by the Buell Combustion Company. As shown in Fig. 23.15, the wet solid is fed in a thin layer to the top member of a series of annular shelves each made of a number of segmental plates with slots between them. These shelves rotate and by means of suitably placed arms the material is pushed through a slot on to a shelf below; after repeated movements, the solid leaves at the bottom of the dryer. The shelves are heated by a row of steam pipes, and in the centre there are three or more fans which suck the hot air over the material and remove it at the top.

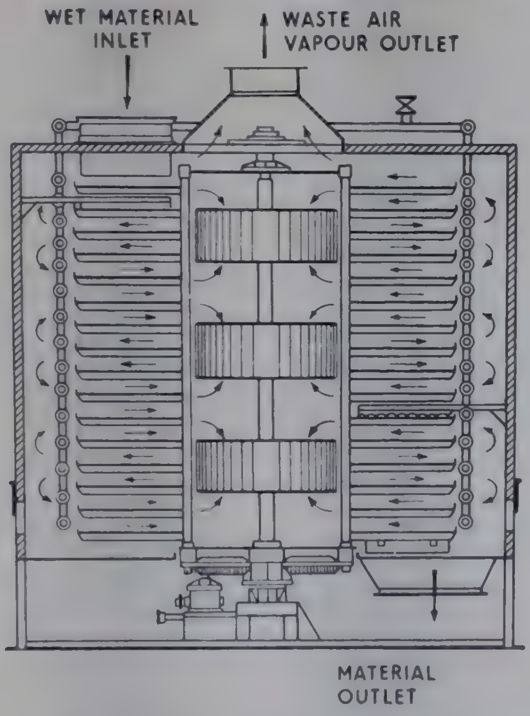


Fig. 23.15. Turbo-shelf dryer

### Freeze Drying

In this process, the material is first frozen and then dried by sublimation in a very high vacuum of the order of 0·1 to 0·3 mm of mercury, at a temperature in the range of −10 to −30°C. During the sublimation of the ice, a dry surface layer is left; this is not free to move because it has been frozen, and a honeycomb structure is formed. With normal vacuum drying of biological solutions containing dissolved salts, a high local salt concentration is formed at the skin, but with freeze drying this does not occur because of the freezing of the solid. Thus, freeze drying is useful not only as a method of working at low temperatures, but also as a method of avoiding surface hardening.

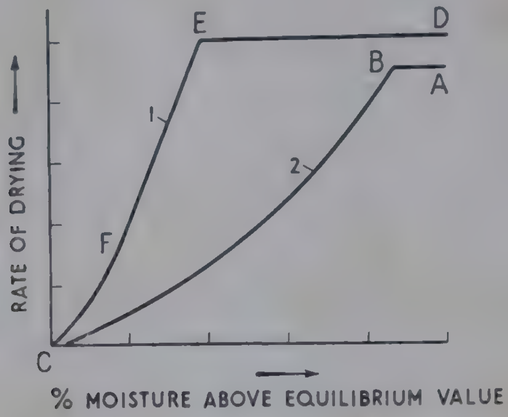


Fig. 23.16. Rate of drying of solid material (granular)

As indicated by CHAMBERS[21], heat has to be supplied to the material to provide the heat of sublimation. The rate of supply of heat should be such that the highest possible water vapour pressure is obtained at the ice surface without danger of melting the material. During this stage, well over 95% of the water present should be removed, and to complete the drying the material should be allowed to rise in temperature, to say room temperature. The great attraction of this technique is that it does not harm heat-sensitive materials, and it has been widely used for the drying of penicillin, and other biological materials. The main disadvantage is the high cost of the process.

## RATE OF DRYING

In the drying of material, we have to remove some free moisture from the surface and also moisture from the interior. If we determine the change in moisture content with time for a material, we obtain a smooth curve from which the rate of drying at any given moisture content can be obtained. The form of the drying rate curve varies with the structure and type of material, but two typical curves are illustrated in Fig. 23.16. In curve (2) there are two well-defined zones: *AB*, where the rate of drying is constant; and *BC*, where there is a steady fall in the rate of drying as the percentage of moisture is reduced. The moisture content at the end of the constant rate period is represented by point *B*, and is known as the critical moisture content. Curve (1) which will be obtained for a rather different kind of material shows three stages: *DE*, *EF* and *FC*. The stage *DE* represents a constant rate period, and *EF* and *FC*

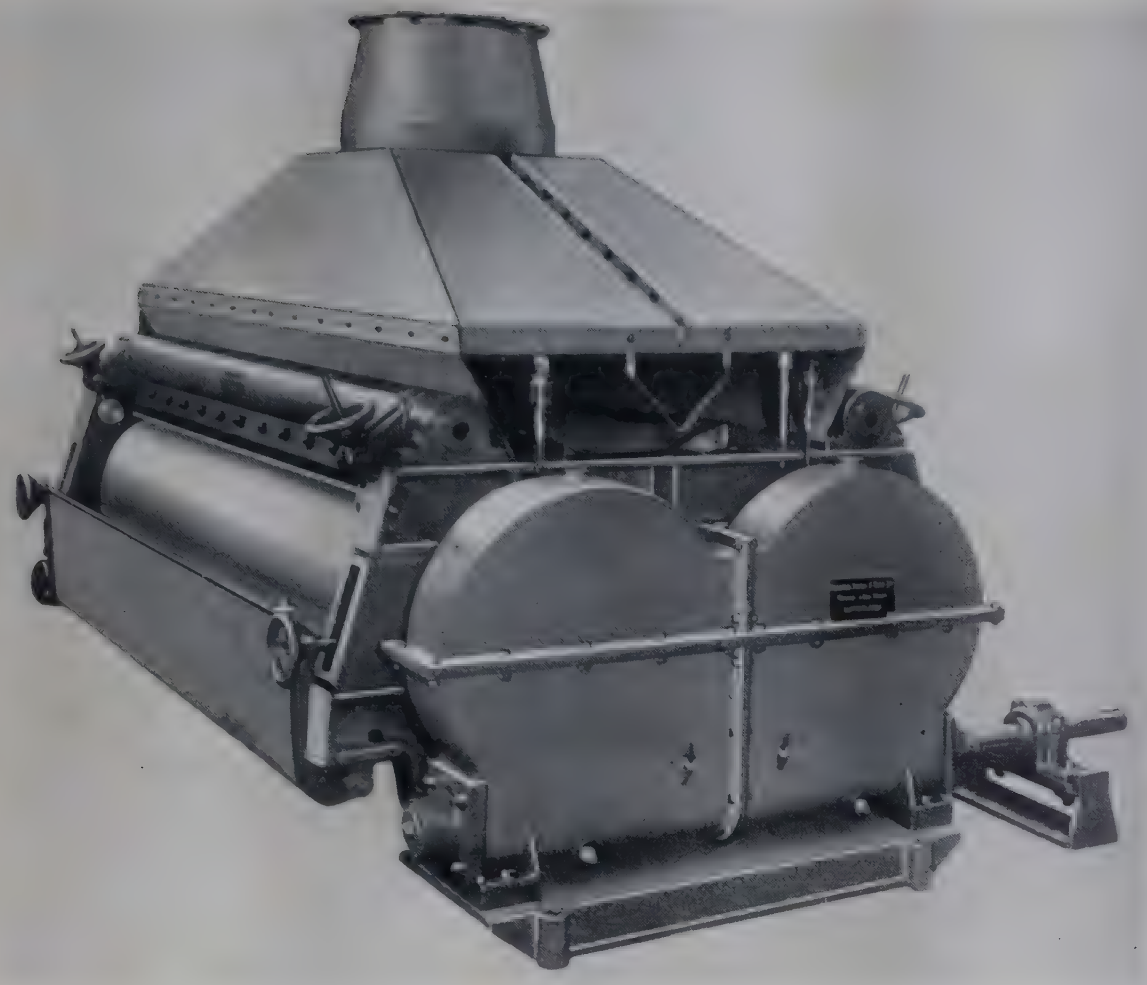
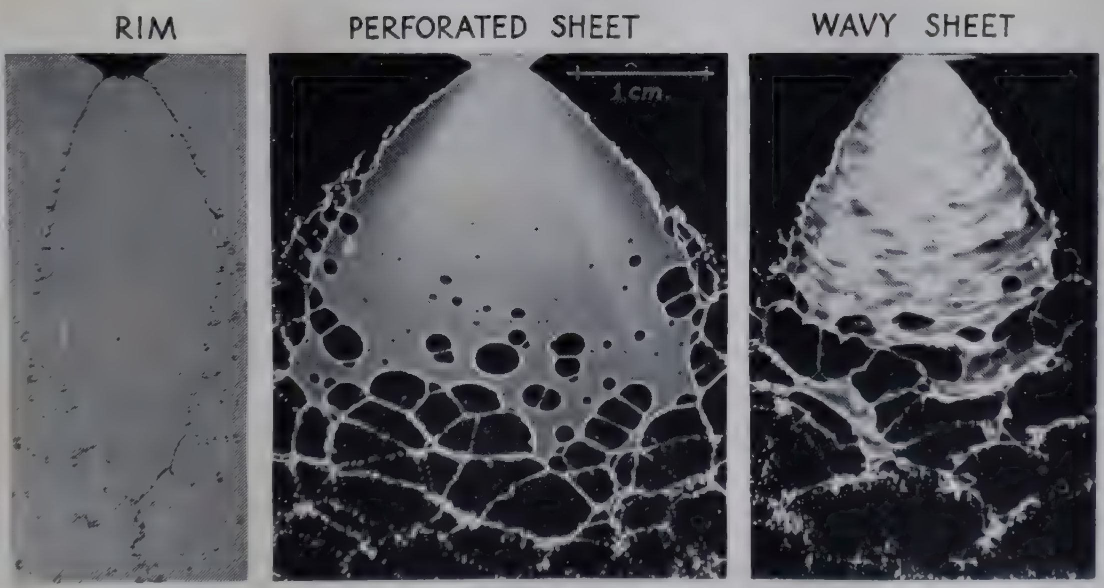


Fig. 23.17. Double drum dryer, 36 in. diameter × 100 in. long



RIM

PERFORATED SHEET

WAVY SHEET

Fig. 23.18. Modes of disintegration of liquid films[22]

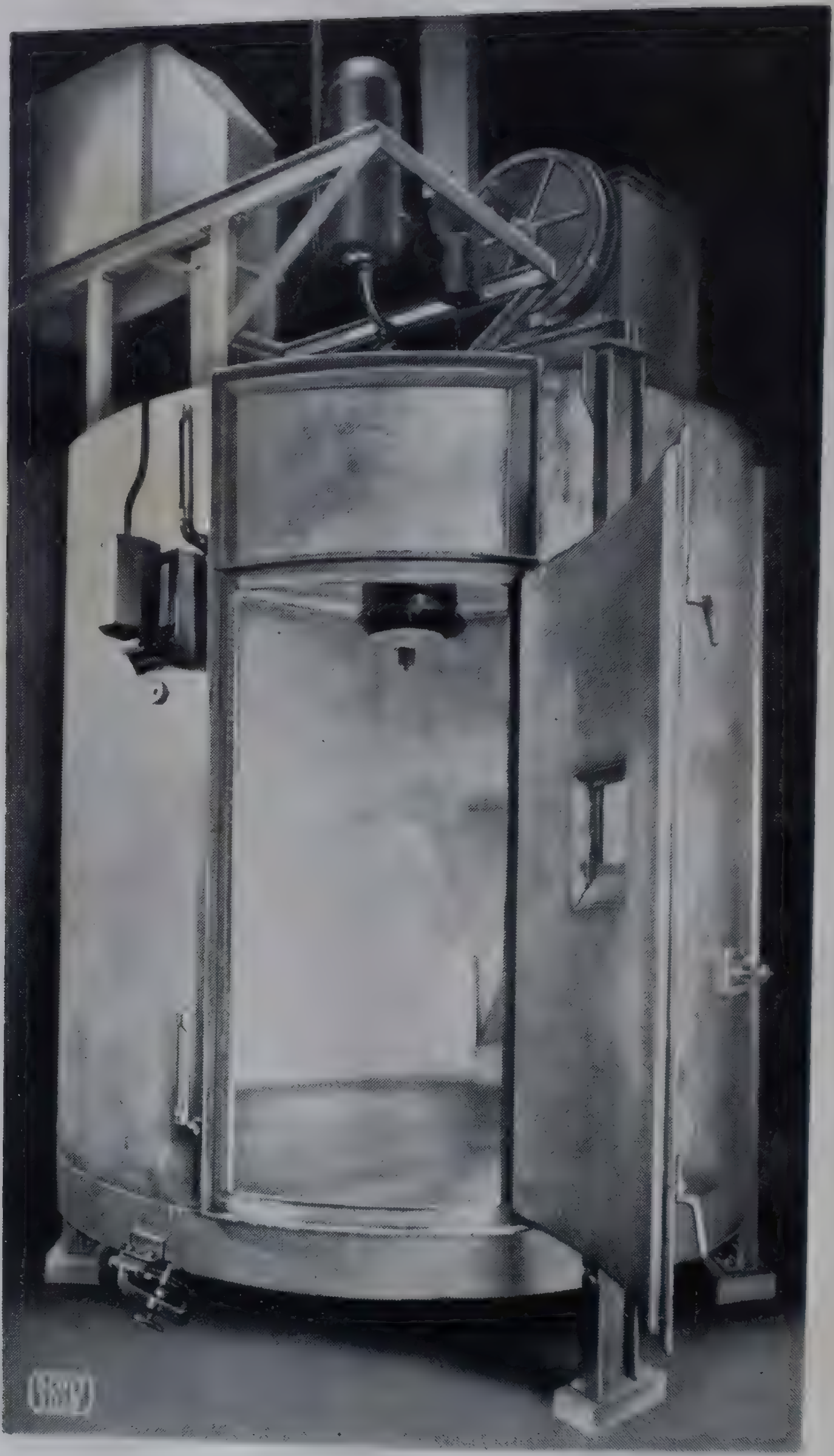


Fig. 23.19. Semi-industrial size spray dryer

are falling rate periods. In this case, however, the section *EF* is a straight line and only the portion *FC* is curved; section *EF* is known as the first falling rate period and the final stage, shown as *FC*, as the second falling rate period. The drying of soap gives rise to a curve of type (2), and sand to a curve of type (1). A number of workers, notably SHERWOOD(9) and NEWITT and co-workers(20), have made contributions to the theories on the rate of drying in these various stages.

## Constant Rate Period

During the constant rate period, it is assumed that drying takes place from a saturated surface of the material by diffusion of the water vapour through a stationary air film into the air stream. Thus, GILLILAND(12) has shown that the rates of drying of a variety of materials in this stage are substantially the same (Table 23.3).

TABLE 23.3. *Evaporation rates for various materials under constant conditions* (GILLILAND)

| *Material* | *Rate of evaporation gm/hr-sq. cm.* |
|---|---|
| Water | 0·27 |
| Whiting pigment | 0·21 |
| Brass filings | 0·24 |
| Brass turnings | 0·24 |
| Sand (fine) | 0·20–0·24 |
| Clays | 0·23–0·27 |

To calculate the rate of drying under these conditions, we make use of the relationships previously obtained for diffusion of a vapour from a liquid surface into a gas. The simplest equation of this type would be

$$W = K_G A(P_s - P_w) \qquad \frac{dw}{dt} = -KA(P_s - P_W) \qquad \ldots .(23.11)$$

where $K_G$ represents the diffusional transfer coefficient.
Since the rate of transfer will depend on the velocity $u$ of the air stream, raised to a power of about 0·8, we can write

$$W = K' A(P_s - P_w)u^{0 \cdot 8} \qquad \ldots .(23.12)$$

In the above equations,

$A$ is the surface area,
$P_s$ is the vapour pressure of the water,
$P_w$ is the partial pressure of water vapour in the air stream, and
$W$ is the mass rate of evaporation.

This type of equation has been used in Volume 1 for studying the rate of vaporisation into an air stream, and simply states that the rate of transfer is equal to the transfer coefficient multiplied by the driving force. It should be noted, however, that $P_s - P_w$ is not only a driving force, but is also related to the capacity of the air stream to absorb moisture.

These equations suggest that the rate of drying is independent of the geometrical shape of the surface. Experiments by POWELL and GRIFFITHS(8) have shown, however, that the ratio of the length to the width of the surface is of some importance, and that the evaporation rate (gm/sec) is given more accurately by equations 23.13 and 23.14, below.

(1) For values of $u$ between 100 and 300 cm/sec,

$$W = 2{\cdot}12 \times 10^{-7} L^{0{\cdot}77} B (P_s - P_w)(1 + 0{\cdot}121 u^{0{\cdot}85}) \qquad \ldots .(23.13)$$

(2) For values of $u$ below 100 cm/sec,

$$W = 4{\cdot}3 \times 10^{-7} L^{0{\cdot}73} B^{0{\cdot}8} (P_s - P_w)(1 + 0{\cdot}121 u^{0{\cdot}85}) \qquad \ldots .(23.14)$$

where $P_s$, the saturation pressure at the temperature of the surface, and $P_w$, the vapour pressure in the air stream, are both in mm of mercury.

$L$ and $B$ are the length and width of the surface in cm.

For all normal engineering design work we may take it that the rate of drying is proportional to the transfer coefficient multiplied by $P_s - P_w$. CHAKRAVORTY'S(17) work at the Imperial College has shown that, if the temperature of the surface is greater than that of the air stream, then $P_w$ may easily reach a value corresponding to saturation of the air. Under these conditions, the capacity of the air to take up moisture is zero, while the force causing evaporation is $P_s - P_w$. As a result, a mist will form and water may be redeposited on the surface. In all drying equipment, care must therefore be taken to see that the air or gas used does not become saturated with moisture.

The rate of drying in the constant rate period will be given by

$$\boxed{\frac{\mathrm{d}w}{\mathrm{d}t} = \frac{hA\Delta T}{\lambda} = K_G A (P_s - P_w)} \qquad \ldots .(23.15)$$

where $\frac{\mathrm{d}w}{\mathrm{d}t}$ is the rate of loss of water [in British units, lb/hr],

$h$ is the heat transfer coefficient from air to the wet surface,

$\Delta T$ is the temperature difference between the air and the surface,

$\lambda$ is the latent heat of vaporisation,

$K_G$ is the mass transfer coefficient for diffusion from the wet surface through the gas film,

$A$ is the area of interface for heat and mass transfer, and

$P_s - P_w$ is the difference between the vapour pressure of water at the surface and the partial pressure in the air.

It is rather more convenient to express the mass transfer coefficient in terms of a humidity difference, so that $K_G A(P_s - P_w) \simeq kA(\mathscr{H}_s - \mathscr{H})$. The rate of drying is thus determined by the values of $h$, $\Delta T$ and $A$, and is not influenced by the conditions inside the solid. $h$ will depend on the air velocity and the direction of flow of the air, and it has been found that $h = C \,.\, G'^{0{\cdot}8}$ where $G'$

is the mass rate of flow of air in lb/hr-ft². For air flow parallel to plane surfaces, SHEPHERD et al.[11] have given the value of $C$ as 0·0128, for the heat transfer coefficient expressed in lb-cal/hr-ft²-°C (B.Th.U/hr-ft²-°F).

If the gas temperature is high, then a considerable proportion of the heat will pass to the solid by radiation, and the heat transfer coefficient will increase. This may result in the temperature of the solid rising above the wet bulb temperature.

### First Falling Rate Period

The points $B$ and $E$ in Fig. 23.16 represent conditions where the surface is no longer capable of supplying sufficient free moisture to saturate the air in contact with it. Under these conditions, the real factor influencing the rate of drying is the mechanism by which the moisture from inside the material is transferred to the surface. In general, the curves in Fig. 23.16 will apply, but for type 2 we can obtain a simplified expression for the rate of drying in this period.

### Second Falling Rate Period

At the conclusion of the first falling rate period we may assume that the surface is dry and that the plane of separation is moving into the solid. In this case, evaporation will be taking place from within the solid, the vapour reaching the surface by molecular diffusion through the material. The forces controlling the vapour diffusion will determine the final rate of drying, and these will be largely independent of the conditions outside the material.

### Time for Drying

Suppose a material to be dried by passing hot air over the surface which is initially wet. Then the rate of drying curve in its simplest form is represented by $BCE$ as in Fig. 23.20.

Here $w$ is the total moisture,

$w_e$ is the equilibrium moisture content ($E$),

$f$ is the free moisture content ($w - w_e$), and

$w_c$ is the critical moisture content.

***Constant Rate Period*** During the period of drying from the initial moisture content $w_1$ to the critical moisture content $w_c$, the rate of drying is constant, so that the time of drying $t_c$ is given by

$$t_c = \frac{w_1 - w_c}{R_c A} \qquad \text{....(23.16)}$$

where $R_c$ is the rate of drying per unit area in the constant rate period, and $A$ is the area of exposed surface.

***Falling Rate Period.*** During this period the rate of drying is directly proportional to the free moisture content $w - w_e$

i.e. $$-\frac{1}{A} \cdot \frac{dw}{dt} = m(w - w_e) = mf$$

$$\therefore \quad -\frac{1}{mA}\int_{w_c}^{w} \frac{dw}{w - w_e} = \int_0^{t_f} dt$$

$$\therefore \quad \frac{1}{mA} \cdot \ln\left[\frac{w_c - w_e}{w - w_e}\right] = t_f$$

$$\therefore \quad t_f = \frac{1}{mA} \cdot \ln\frac{f_c}{f} \qquad \ldots.(23.17)$$

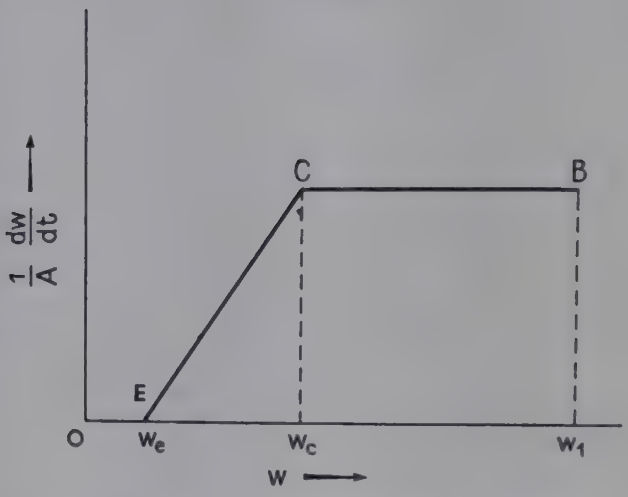


Fig. 23.20. Use of rate of drying curve for estimation of time of drying

Total time of drying from $w_1$ to $w$ is $t = t_c + t_f$.

The rate of drying $R_c$ over the constant rate period is equal to the initial rate of drying in the falling rate period, so that $R_c = mf_c$.

$$\therefore \quad t_c = \frac{w_1 - w_c}{mf_c A} \qquad \ldots.(23.18)$$

∴ Total drying time $$t = \frac{w_1 - w_c}{Amf_c} + \frac{1}{mA}\ln . \frac{f_c}{f}$$

$$= \frac{1}{mA}\left[\frac{f_1 - f_c}{f_c} + \ln\frac{f_c}{f}\right] \qquad \ldots.(23.19)$$

## THE MECHANISM OF MOISTURE MOVEMENT DURING DRYING

The general form of the curve for the rate of drying of a solid has been given (Fig. 23.16), and it has been shown that this takes the form of two or sometimes three distinct sections. During the constant rate period, moisture vaporises into the air stream and the controlling factor is the transfer coefficient for diffusion across the gas film. It is important to understand how the moisture moves to the drying surface during the falling rate period. Two ideas have been given of the physical nature of this process, one the diffusion theory and the second the capillary theory. In the diffusion theory, it is supposed that the rate of movement of water to the air interface is governed by rate equations similar to those for heat transfer, whilst in the capillary theory it is assumed that the forces controlling the movement of water are capillary in origin and arise from the minute pore spaces between the individual particles.

### Falling Rate Period. Diffusion Control

In the falling rate period, the surface is no longer completely wetted and the rate of drying steadily falls. In the analysis above, it has been assumed that the effective wetted area is a linear function of the water content, so that the rate of drying is given by

$$\frac{1}{A} \cdot \frac{\mathrm{d}w}{\mathrm{d}t} = -m[w - w_e] \qquad \ldots.(23.20)$$

In many instances, however, the rate of drying is governed by the rate of internal movement of the moisture to the surface. It was initially assumed that this movement was a process of diffusion and would follow the same laws as heat transfer. This approach has been examined by a number of workers and in particular by SHERWOOD⁽⁴⁾ and NEWMAN⁽⁵⁾.

Let $w$ be the liquid content of the solid,

$w_1$ be the initial content, and

$w_e$ be the equilibrium content.

Then $\dfrac{w - w_e}{w_1 - w_e} = \dfrac{\text{Free liquid content at any point at any time}}{\text{Initial free liquid content}}$

Consider a slab with the edges coated to prevent evaporation, and suppose the slab to be dried by evaporation from two opposite faces, the $Y$-direction being taken perpendicular to the drying face, the central plane being taken as $y = 0$, and the slab thickness $2l$. Then, on drying, the moisture movement by diffusion will be in the $Y$-direction and we have:

$$\frac{\mathrm{d}w}{\mathrm{d}t} = D_L \frac{\mathrm{d}^2 w}{\mathrm{d}t^2} \qquad \ldots.(23.21)$$

where $D_L$ is the coefficient of diffusion for the liquid (in British units, ft²/hr). Then, SHERWOOD and NEWMAN have given the solution of this equation in the form:

$$\frac{w - w_e}{w_1 - w_e} = \frac{8}{\pi^2}\left\{e^{-D_L t\left(\frac{\pi}{2l}\right)^2} + \tfrac{1}{9}e^{-9D_L t\left(\frac{\pi}{2l}\right)^2} + \tfrac{1}{25}e^{-25D_L t\left(\frac{\pi}{2l}\right)^2} + \ldots\right\} \qquad \ldots.(23.22)$$

It should be noted that this equation assumes an initial uniform distribution of moisture, and that the drying is from both surfaces. When drying is from one surface, then $l$ is the total thickness. If the time of drying is long, then only the first term of the equation need be used and we get, on differentiation of equation 23.22:

$$\frac{\mathrm{d}w}{\mathrm{d}t} = -\frac{\pi^2 D_L}{4l^2}(w - w_e) \qquad \ldots.(23.23)$$

In the drying of materials such as wood or clay, the moisture content at the end of the constant rate period is not uniform, but is more nearly parabolic. SHERWOOD has developed an analysis for this case, and has given experimental values for the drying of brick clay.

In this solution it is assumed that the rate of movement of water is proportional to a concentration gradient, and capillary and gravitational forces are neglected. Water may, however, flow from regions of low concentration to those of high concentration if the pore sizes are suitable, and for this and other reasons CEAGLSKE and HOUGEN(10) suggested the capillary theory which is briefly outlined in the next section.

### Capillary Theory of Drying

The capillary theory of drying has been put forward to provide a means of explaining the movement of moisture in the bed during surface drying. The basic importance of the pore space between granular particles was first pointed out by SLICHTER(2) in connection with the movement of moisture in soils, and this work has now been modified and considerably expanded by HAINES(9); the principles will be given and then applied to the problem of drying. Imagine a systematic packing of uniform spherical particles. These may be arranged in six different regular ways, ranging from the most open to the closest packing. In the former, the spheres are arranged as if at the corners of a cube, each sphere touching six others; whilst in the latter arrangement, each sphere rests in the hollow of three spheres in adjacent layers, and touches twelve other

TABLE 23.4. *Some properties of packings of spheres of radius r*

| *Packing arrangement* | *Pore space % total volume* | *Radius of pore* | *Radius of waist* | *Value of "x" in equation 23.27 for:* | |
|---|---|---|---|---|---|
| | | | | *Limiting suction potential of pores* | *Entry suction potential of waists* |
| Cubical | 47·64 | 0·700$r$ | 0·414$r$ | 2·86 | 4·82 |
| Rhombohedral | 25·95 | 0·288$r$ | 0·155$r$ | 6·90 | 12·90 |

spheres. These configurations are shown in Fig. 23.21. The densities of packing of the other four arrangements will lie between those illustrated.

In each case, a regular group of spheres surrounds a space which is called a pore, and the bed is made up of a series of these elemental groupings. The pores are connected together by passages of various sizes, the smallest portions of which are known as waists. The size of a pore is defined as the diameter of the largest sphere which can be fitted into it, and the size of a waist as the diameter of the inscribed circle. The sizes of the pores and waists will differ for each form of packing, as indicated in Table 23.4.

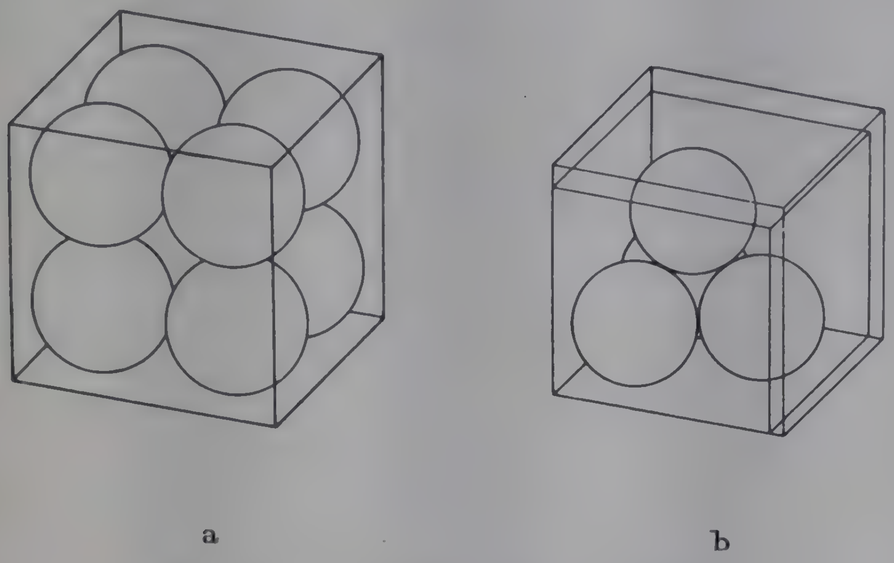


Fig. 23.21. Packing of spherical particles

(*a*) Cubic arrangement, one sphere touching six others

(*b*) Rhombohedral packing, one sphere touching twelve others, with layers arranged in rhombic formation

The continuous variation in the diameter of each passage is the essential difference between a granular packing and a series of capillary tubes. If a clean capillary of uniform diameter $2r'$ is placed in a liquid, the level will rise in the capillary to a height $h_s$ given by equation 23.24.

$$h_s = \frac{2\sigma}{r'\rho g}\cos\alpha \qquad \ldots.(23.24)$$

where $\rho$ is the density of the liquid,

$\sigma$ is the surface tension,

$\alpha$ is the angle of contact, and

$g$ is the acceleration due to gravity.

and a negative pressure, known as a suction potential, will exist in the liquid in the capillary. Immediately below the meniscus the suction potential will be equivalent to the height of the liquid column $h_s$ and, if water is used, will have the value:

$$h_s = \frac{2\sigma}{r'\rho g} \qquad \ldots.(23.25)$$

If equilibrium conditions exist, the suction potential $h_1$ at any other level in the liquid, a distance $z_1$ below the meniscus, will be given by the equation:

$$h_s = h_1 + z_1 \qquad \text{....(23.26)}$$

Similarly, if a uniform capillary is filled to a greater height than $h_s$, as given by equation 23.24, and its lower end is immersed, the liquid column will recede to this height.

The non-uniform passages in a porous material will also display the same characteristics as a uniform capillary, with the important difference that the rise of water in the passages will be limited by the pore size, whilst the depletion of saturated passages will be controlled by the size of the waists. The height of rise is controlled by the pore size, since this approximates to the largest section of a varying capillary, whilst the depletion of water is controlled by the narrow waists which are capable of a higher suction potential than the pores.

The theoretical suction potential of a pore or waist containing water can therefore be calculated from the equation

$$h_t = \frac{x\sigma}{r\rho g} \qquad \text{....(23.27)}$$

where $x$ is a factor depending on the type of packing, and
$r$ is the radius of the spheres.

For example:

For an idealised bed of uniform rhombohedrally packed spheres of radius $r$, the waists are of radius $0{\cdot}155r$ (from Table 23.4).

The maximum theoretical suction potential of which such a waist is capable

$$= \frac{2\sigma}{0{\cdot}155r\rho g}$$

$$= \frac{12{\cdot}9\sigma}{r\rho g},$$

from which it is seen $x = 12{\cdot}9$ in this instance.

The maximum suction potential that can be developed by a waist is known as the "entry" suction potential. This is the controlling potential required to open a saturated pore protected by a meniscus in an adjoining waist. Some values for $x$ are given in Table 23.4.

When a bed is composed of granular material with particles of mixed sizes, the suction potential cannot be calculated but must be measured by methods such as those given by HAINES(3) and NEWITT *et al.*(20).

***Drying of a granular material according to capillary theory.*** Suppose a bed of uniform spheres to be initially saturated and to be surface dried in a current of air of constant temperature, velocity and humidity. Then the rate of drying is given by

$$\frac{\mathrm{d}w}{\mathrm{d}t} = K_G A(P_{w_0} - P_w) \qquad \text{....(23.28)}$$

where $P_{w_0}$ is the saturation partial pressure of water vapour at the wet bulb temperature of the air, and $P_w$ is the partial pressure of the water vapour in the air stream. This rate, as indicated earlier, will remain constant so long as the inner surface of the "stationary" air film remains saturated.

As evaporation proceeds, the water surface recedes into the waists between the top layer of particles, and an increasing suction potential is developed in the liquid. When the menisci on the cubical waists (i.e. the largest) have receded to the narrowest section, the suction potential $h_s$ at the surface equals $\frac{4{\cdot}82\sigma}{r\rho g}$ (Table 23.4). Further evaporation will result in $h_s$ increasing so that the menisci on the surface cubical waists will collapse, and the larger pores below will open. As $h_s$ steadily increases, the entry suction of progressively finer surface waists is reached, so that the menisci collapse into the adjacent pores which are thereby opened.

In considering the conditions below the surface, the suction potential $h_1$ a distance $z_1$ from the surface will be given by equation 23.26.

$$h_s = h_1 + z_1 \qquad \text{....(23.26)}$$

The flow of water through waists surrounding an open pore will be governed by the size of the waist in the following way:

(1) If the size of the waist is such that its entry suction potential exceeds the suction potential at that level within the bed, it will remain full by the establishment of a meniscus therein, in equilibrium with the effective suction potential to which it is subjected. This waist will then protect adjoining full pores which cannot be opened until one of the waists to which it is connected collapses.

(2) If the size of the waist is such that its entry suction potential is less than the suction potential existing at that level, it will in turn collapse and open the adjoining pore. Moreover, this successive collapse of pores and waists will progressively continue, so long as the pores so opened expose waists having entry suction potentials less than the suction potentials existing at that depth.

As drying proceeds, two processes take place simultaneously:

(1) The collapse of progressively finer surface waists, and the resulting opening of pores and waists connected thereto, which they previously protected.

(2) The collapse of further full waists within the bed adjoining opened pores, and the consequent opening of adjacent pores.

It should be noted that, even though the effective suction potential at a waist or pore within the bed may be in excess of its entry or limiting suction potential this will not necessarily collapse or open. Such a waist can only collapse if i adjoins an opened pore, and the pore in question can only open upon the collapse of an adjoining waist.

*Effect of Particle Size.* Reducing the particle size of the bed will reduce the size of the pores and the waists, and will increase the entry suction potential of the waists. This increase will mean that the percentage variation in suction potentials with depth will be reduced, and the moisture distribution will be more uniform with small particles.

As the pore sizes are reduced, the frictional forces opposing the movement of water through these pores and waists may become significant, so that equation 23.26 is more accurately represented by

$$h_s = h_1 + z_1 + h_f \qquad \ldots .(23.29)$$

where $h_f$ is the frictional head opposing the flow over a depth $z_1$ from the surface. The value of $h_f$ will depend on the particle size and it has been found(20) that with coarse particles when only low suction potentials are found, the gravity effect is important but $h_f$ is small, whilst with fine particles $h_f$ becomes large. It has been found that:

(1) For particles of between $10^{-1}$ and $10^{-2}$ cm radius, the values of $h_1$ are independent of the rate of drying, but vary appreciably with depth. Frictional forces are, therefore, negligible; capillary and gravitational forces are in equilibrium throughout the bed and are the controlling forces. Under such circumstances the percentage moisture loss at the critical point at which the constant rate period ends is independent of the drying rate, but varies with the depth of bed.

(2) For particles of between $10^{-3}$ and $10^{-4}$ cm radius, the values of $h_1$ vary only slightly with rate of drying and depth, indicating that both gravitational and frictional forces are negligible; capillary forces are controlling. The critical point will here be independent of drying rate and depth of bed.

(3) For particles of less than $10^{-4}$ cm radius, gravitational forces are negligible, whilst frictional forces are of increasing importance; capillary and frictional forces may then be controlling. In such circumstances, the percentage moisture loss at the critical point will diminish with increased rate of drying and depth of bed. With beds of very fine particles, an additional factor comes into play. The very high suction potentials which are developed cause a sufficient reduction of the pressure for vaporisation of water to take place inside the bed. This internal vaporisation results in a breaking up of the continuous liquid phase and a consequent interruption in the free flow of liquid by capillary action. Hence, the rate of drying is still further reduced.

Some of the experimental results of NEWITT *et al.*(20) are illustrated in ig. 23.22.

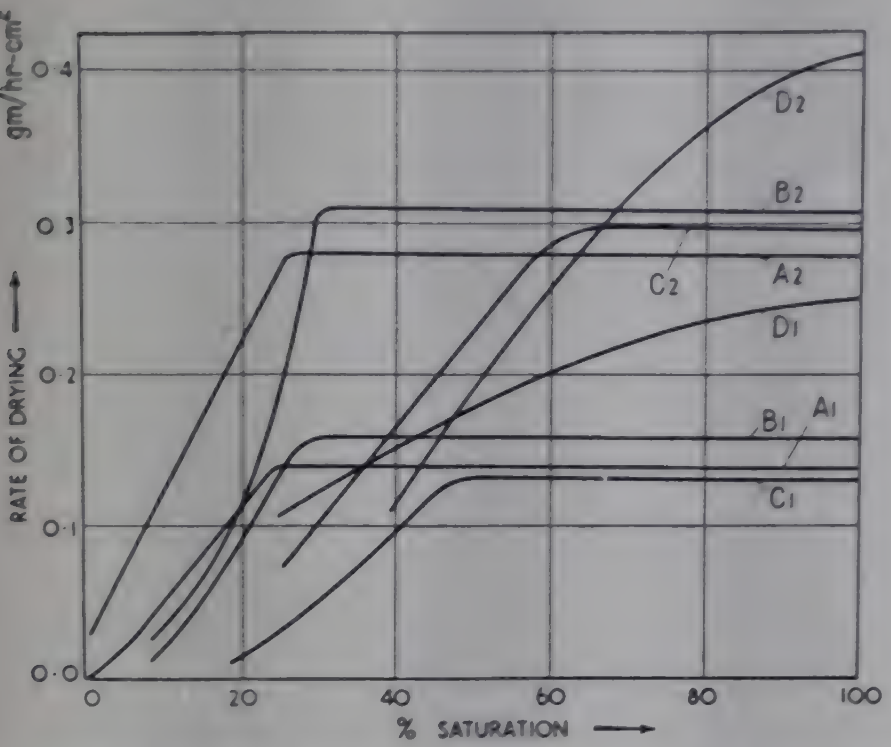


Fig. 23.22. Rates of drying of various materials as a function of percentage saturation

*A* $60 \times 10^{-4}$ cm glass spheres; bed 5·1 cm deep
*B* $23{\cdot}5 \times 10^{-4}$ cm silica flour; bed 5·1 cm deep
*C* $7{\cdot}5 \times 10^{-4}$ cm silica flour; bed 5·1 cm deep
*D* $2{\cdot}5 \times 10^{-4}$ cm silica flour; bed 6·5 cm deep
1. Low drying rate
2. High drying rate

## DRYING OF GASES

The drying of gases is carried out on a very large scale, the most important applications of the process being as follows:

(1) Drying gas in order to reduce its tendency to cause corrosion.

(2) Preparation of dry gas for use in a chemical reaction.

(3) Reduction of the humidity of air in air-conditioning plants.

The problems involved in the drying or dehumidification of gases have already been referred to in Chapter 10 of Volume 1. The most important methods available will now be summarised.

*Cooling*. A gas stream may be dehumidified by bringing it into contact with a cold liquid or a cold solid surface. If the temperature of the surface is lower than the dew point of the gas, condensation will take place, and the temperature of the surface will tend to rise by virtue of the liberation of latent heat; it is therefore necessary constantly to remove heat from the surface. Because a far larger interfacial surface can be produced with a liquid, it is usual to spray a liquid into the gas and then to cool it again before it is recycled. In many cases, countercurrent flow of the gas and liquid is obtained by introducing the liquid at the top of a column and allowing the gas to pass upwards.

*Compression.* The humidity of a gas may be reduced by compressing it, cooling it down to near its original temperature, and then draining off the water which has condensed. During compression, the partial pressure of the water vapour increases and condensation occurs as soon as the saturation value is exceeded. Coal gas is frequently compressed before it is circulated in order to reduce the amount of corrosion in the distributing mains.

*Liquid absorbents.* If the partial pressure of the water in the gas is less than the equilibrium partial pressure at the surface of a liquid, condensation will take place as a result of contact between the gas and liquid. Thus, water vapour is frequently removed from a gas by bringing it into contact with concentrated sulphuric acid, phosphoric acid, or glycerol. Concentrated solutions of salts, such as calcium chloride, are also effective. The process may be carried out either in a packed column or in a spray chamber. Regeneration of the liquid is an essential part of the process, and this is usually effected by evaporation.

*Solid absorbents.* The use of silica gel or solid calcium chloride to remove water vapour from gases is a common operation in the laboratory. Moderately large units can be made, but the volume of packed space required is generally large because of the comparatively small transfer surface per unit volume. If the particle size is too small, the pressure drop through the material becomes excessive. The solid desiccants are also regenerated by heating.

Coal gas is frequently dried by using a calcium chloride liquor containing about 5·6 pounds of calcium chloride per gallon. The extent of recirculation of the liquor from the base of the tower is governed by heating effects, since the condensation of the water vapour gives rise to considerable heating. It is necessary to fit heat exchangers to cool the liquor leaving the base of the tower.

In the contact plant for the manufacture of sulphuric acid, sulphuric acid is itself used for drying the air for the oxidation of the sulphur. When drying hydrocarbons such as benzene, it is sometimes convenient to pass the material through a bed of solid caustic soda, but if the quantity is appreciable this method will be expensive.

The great advantage of the materials such as silica gel and activated alumina is that they enable the gas to be almost completely dried. Thus, with silica gel, air may be dried down to a dew point of —94°F. Small silica gel containers are frequently used to prevent moisture condensation in the low pressure lines of pneumatic control installations.

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## LIST OF SYMBOLS USED IN CHAPTER 23

| | | |
|---|---|---|
| $A$ | Area for heat transfer or evaporation | $L^2$ |
| $a$ | Surface area per unit volume | $L^{-1}$ |
| $B$ | Width of surface | $L$ |
| $C$ | Coefficient | $M^{0.2}L^{-0.4}T^{-0.2}$ |
| $d_m$ | Main drop diameter | $L$ |
| $D$ | Diameter of drum or disc | $L$ |
| $D_L$ | Diffusion coefficient | $L^2T^{-1}$ |
| $F$ | Volumetric rate of feed per unit cross-section | $LT^{-1}$ |
| $f$ | Free moisture content | $M$ |
| $f_c$ | Free moisture content at critical condition | $M$ |
| $G'$ | Mass rate of flow of air per unit cross-section | $ML^{-2}T^{-1}$ |
| $g$ | Acceleration due to gravity | $LT^{-2}$ |
| $\mathscr{H}$ | Humidity | — |
| $\mathscr{H}_0$ | Humidity of saturated air | — |
| $\mathscr{H}_s$ | Humidity of saturated air at surface temperature | — |
| $h$ | Heat transfer coefficient | $ML^{-2}T^{-1}$ |
| $h_D$ | Mass transfer coefficient | $LT^{-1}$ |
| $h_f$ | Friction head over a distance $z_1$ from surface | $L$ |
| $h_s$ | Suction potential immediately below meniscus | $L$ |
| $h_t$ | Theoretical suction potential of pore or waist | $L$ |
| $h_1$ | Suction potential at distance $z_1$ below meniscus | $L$ |
| $K$ | Coefficient | $M^{-1}L^2T$ |
| $K'$ | Coefficient | $L^{-1.8}T^{1.8}$ |
| $K''$ | Transfer coefficient ($h_D\rho_A$) | $ML^{-2}T^{-1}$ |
| $K_G$ | Diffusion coefficient | $L^{-1}T$ |
| $k$ | Coefficient | $ML^{-2}T^{-1}$ |
| $L$ | Length of surface | $L$ |
| $l$ | Half thickness of slab | $L$ |
| $M_A$ | Molecular weight of air | — |
| $M_w$ | Molecular weight of water | — |
| $m$ | Ratio of rate of drying per unit area to moisture content | $L^{-2}T^{-1}$ |
| $N$ | Revolutions per unit time | $T^{-1}$ |
| $n_f$ | Number of flights | — |
| $P$ | Total pressure | $ML^{-1}T^{-2}$ |
| $P_s$ | Vapour pressure of water at surface of material | $ML^{-1}T^{-2}$ |
| $P_w$ | Partial pressure of water vapour | $ML^{-1}T^{-2}$ |
| $P_{w0}$ | Partial pressure at surface of material at wet bulb temperature | $ML^{-1}T^{-2}$ |
| $Q$ | Rate of heat transfer | $MT^{-1}\theta$ |
| $R_c$ | Rate of drying per unit area for constant rate period | $ML^{-2}T^{-1}$ |
| $\mathbf{R}$ | Universal gas constant | $L^2T^{-2}\theta^{-1}$ |
| $r$ | Radius of sphere | $L$ |
| $r'$ | Radius of capillary | $L$ |
| $S$ | Slope of drum | — |
| $T$ | Absolute temperature | $\theta$ |
| $\Delta T$ | Temperature difference | $\theta$ |
| $t$ | Time | $T$ |
| $t_c$ | Time of constant rate period of drying | $T$ |
| $t_f$ | Time of drying in falling rate period | $T$ |
| $U$ | Overall heat transfer coefficient | $ML^{-2}T^{-1}$ |
| $u$ | Gas velocity | $LT^{-1}$ |
| $V$ | Volume | $L^3$ |
| $W$ | Mass rate of evaporation | $MT^{-1}$ |
| $w$ | Total moisture | $M$ |

| | | |
|---|---|---|
| $w_c$ | Critical moisture content | M |
| $w_e$ | Equilibrium moisture content | M |
| $X$ | Hold-up of drum | — |
| $X_a$ | Hold-up of drum with air flow | — |
| $x$ | Factor depending on type of packing | — |
| $y$ | Distance in direction of diffusion | L |
| $z_1$ | Distance below miniscus | L |
| $\alpha$ | Angle of contact | — |
| $\lambda$ | Latent heat of vaporisation | $\theta$ |
| $\rho$ | Density of liquid | $ML^{-3}$ |
| $\rho_A$ | Density of air at its mean partial pressure | $ML^{-3}$ |
| $\sigma$ | Surface tension | $MT^{-2}$ |
| $\theta$ | Gas temperature | $\theta$ |
| $\theta_s$ | Surface temperature | $\theta$ |
| $\theta_w$ | Wet bulb temperature | $\theta$ |

## Section I

# *Size Reduction and Classification of Solids, and Mixing*

## CHAPTER 24

# *Size Reduction of Solids*

In the chemical industry, size reduction is usually carried out in order to increase the surface because, in most reactions involving solid particles, the rate is directly proportional to the area of contact with a second phase. Thus the rate of combustion of solid particles is proportional to the area presented to the gas, though a number of secondary factors may also be involved as, for instance, the free flow of gas may be impeded because of the higher resistance to flow of a bed of small particles. Again in leaching, not only is the rate of extraction increased by virtue of the increased area of contact between the solvent and the solid, but the distance the solvent has to penetrate into the particles in order to gain access to the more remote pockets of solute is also reduced. This factor is also important in the drying of porous solids, where reduction in size causes both an increase in area and a reduction in the distance the moisture must travel within the particles in order to reach the surface; in this case the capillary forces acting on the moisture are also affected.

There are a number of other reasons for effecting size reduction. Thus it may be necessary to break a material into very small particles in order to separate two constituents, especially where one is dispersed in small isolated pockets. Further, the properties of a material may be considerably influenced by the particle size; for example, the chemical reactivity of fine particles is greater than that of coarse particles, and the colour and covering power of a pigment is considerably affected by the size of the particles. Again, far more intimate mixing of solids can be achieved if the particle size is small.

### MECHANISM OF SIZE REDUCTION

The mechanism of the process of size reduction is extremely complex, though in recent years a number of attempts have been made at a more detailed analysis of the problem. If a single lump of material is subjected to a sudden impact, it will generally break so as to yield a few relatively large particles and a number of fine particles, with relatively few particles of intermediate size. If the energy in the blow is increased, the larger particles will be of a rather smaller size and more numerous and, whereas the number of fine particles will be appreciably increased, their size will not be much altered. It therefore appears that the size of the fine particles is closely connected with the internal structure of the material, and the size of the larger particles is more closely connected with the process by which the size reduction is effected.

This effect is well illustrated by a series of experiments on the grinding of coal in a small mill, carried out by HEYWOOD(13). The results are shown in Fig. 24.1 in which the distribution of particle size in the product is shown as a function of the number of revolutions of the mill. The initial size distribution shows a single mode corresponding to a relatively coarse size, but as the degree of crushing is gradually increased this mode progressively decreases in magnitude and a second mode develops at a particular size. This process continues until the first mode has completely disappeared. Here the second mode is characteristic of the material and is known as the persistent mode, and the first is known as the transitory mode.

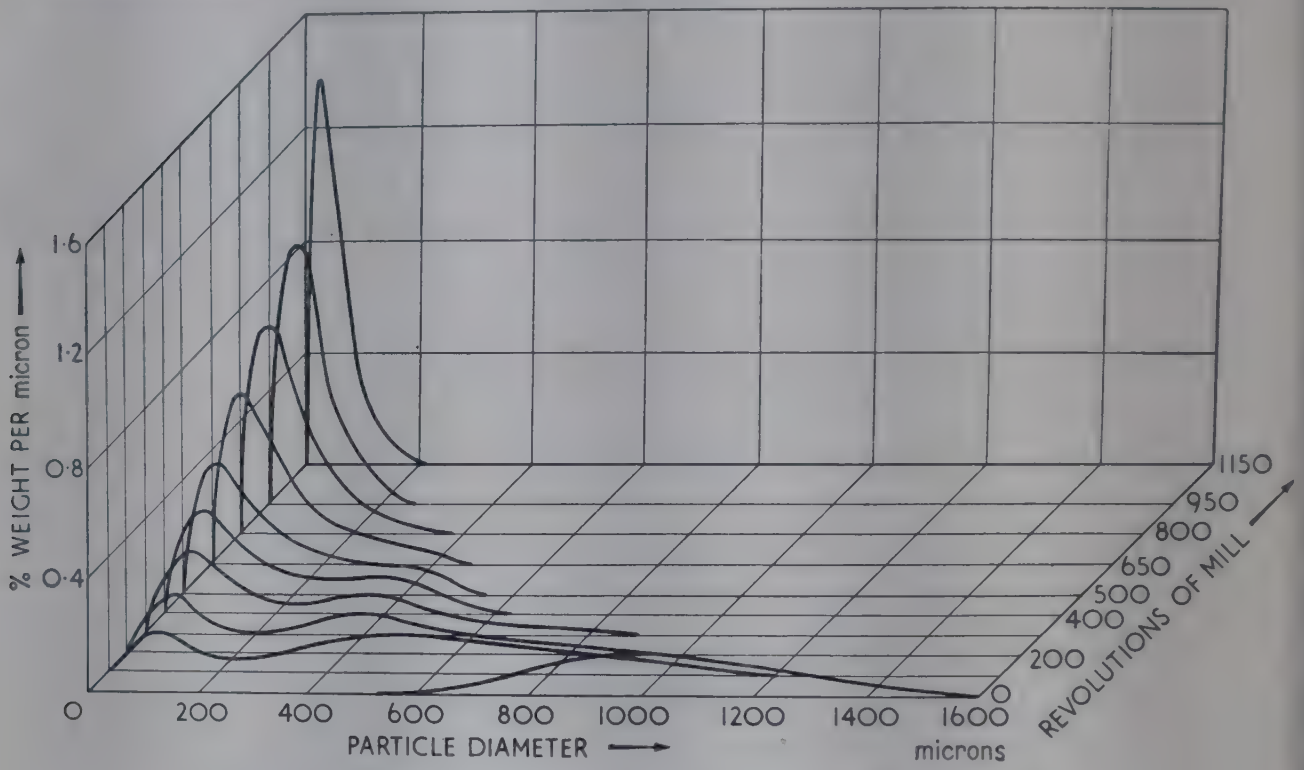

Fig. 24.1. Effect of progressive grinding on size distribution

The energy required to effect size reduction is related to the internal structure of the material and the process really consists of two parts, first opening up any small fissures which are already present and secondly forming new surface. A material such as coal contains a number of small cracks and tends first to break along these, and therefore the large pieces are broken up more readily than the small ones. Further, since a very much greater increase in surface results from crushing a given quantity of fine as opposed to coarse material, fine grinding requires very much more power.

From the point of view of energy utilisation, size reduction is a very inefficient process and only between 0.1 and 2.0% of the energy supplied to the machine appears as increased surface energy in the solids. The efficiency of the process is very much influenced by the manner in which the load is applied and its magnitude, but in addition the nature of the force exerted is also very important, e.g., whether it is predominantly a compressive, an impact or a shearing force. If the applied force is insufficient for the elastic limit to be exceeded, the material is compressed (if compressive force is used) and energy is stored in the particle. When the load is removed, the particle expands again to its original condition without doing useful work; the energy appears as heat and no size reduction is effected. A somewhat greater force, however, will cause the particle to fracture, and in order to obtain the most effective utilisation of energy the force should be only slightly in excess of the crushing strength of the material. The surface of the particles will generally be of a very irregular nature so that the force is initially taken on the high spots with the result that very high stresses and temperatures may be set up locally in the material. As soon as a small amount of breakdown of material takes place, the point of application of the force will alter. The exact method by which fracture occurs is not known but it is suggested by PIRET(20) that the compressive force produces small flaws in the material. If the energy concentration exceeds a certain critical value, these flaws will grow rapidly and will generally branch and the particles will break up. The rate of application of the force is important because there is generally a time lag between attainment of maximum load and fracture. Thus, a rather smaller force will cause fracture provided it is maintained for a sufficient time. This is a phenomenon similar to the ignition lag which is obtained with a combustible gaseous mixture. Here the interval between introducing the ignition source and the occurrence of ignition is a function of the temperature of the source, and when it is near the minimum ignition temperature delays of several seconds may be encountered. The greater the rate at which the load is applied, the less effectively is the energy utilised and the higher is the proportion of fine material which is produced. The efficiency of utilisation of energy as supplied by a falling weight has been compared with that of energy applied slowly by means of hydraulic pressure; up to three or four times more surface can be produced per unit of energy if it is applied by the latter method. PIRET suggests that there is a close similarity between the crushing operation and a chemical reaction. In both cases a critical energy level must be exceeded before the process will start, and in both cases time is an important variable.

## ENERGY FOR SIZE REDUCTION

It is impossible to estimate accurately the amount of energy required in order to effect a size reduction of a given material but a number of empirical laws have been put forward. The two earliest laws were due to KICK(2) and RITTINGER(1) and a third law due to BOND(15, 16) has recently been put forward. These three laws can all be derived from the basic differential equation

$$\frac{\mathrm{d}E}{\mathrm{d}L} = -C \, . \, L^p \qquad \ldots .(24.1)$$

This equation states that the energy $\mathrm{d}E$ required to effect a small change $\mathrm{d}L$ in the size of unit mass of material is a simple power function of the size. If $p$ is put equal to $-2$, integration gives

$$E = C\left(\frac{1}{L_2} - \frac{1}{L_1}\right)$$

Writing $C = K_R f_c$, where $f_c$ is the crushing strength of the material, we obtain RITTINGER'S Law which was first postulated in 1867,

$$E = K_R f_c \left(\frac{1}{L_2} - \frac{1}{L_1}\right) \qquad \ldots .(24.2)$$

Since the surface of unit mass of material is proportional to $\frac{1}{L}$, the interpretation of this law is that the energy required for size reduction is directly proportional to the increase in surface.

On the other hand, if $p$ is put equal to $-1$, we obtain

$$E = C \ln \frac{L_1}{L_2}$$

and writing $C = K_K f_c$

$$E = K_K f_c \ln \frac{L_1}{L_2} \qquad \ldots.(24.3)$$

which is known as KICK's Law. This supposes that the energy required is directly related to the reduction ratio $\frac{L_1}{L_2}$ and that the energy required to crush a given amount of material from a 2 in. to a 1 in. size is the same as that required to reduce the size from $\frac{1}{2}$ in. to $\frac{1}{4}$ in. In equations 24.2 and 24.3, $K_R$ and $K_K$ are known respectively as RITTINGER's constant and KICK's constant. It should be noted that neither is dimensionless.

Neither of the above two laws permits an accurate calculation of the energy requirements. RITTINGER's Law is applicable mainly to that part of the process where new surface is being created and holds most accurately for fine grinding where the increase in surface per unit mass of material is large. KICK's Law, however, more closely relates to the energy required to effect elastic deformation before fracture occurs, and is more accurate than RITTINGER's Law for coarse crushing where the amount of surface produced is considerably less.

BOND has suggested a law intermediate between RITTINGER's and KICK's Laws. Putting $p = -\frac{3}{2}$ in equation 24.1,

$$E = 2C\left(\frac{1}{L_2^{\frac{1}{2}}} - \frac{1}{L_1^{\frac{1}{2}}}\right)$$

$$= 2C\sqrt{\frac{1}{L_2}}\left\{1 - \frac{1}{q^{\frac{1}{2}}}\right\}$$

where $q = \frac{L_1}{L_2}$, the reduction ratio

Writing $C = 5E_i$

$$E = E_i\sqrt{\frac{100}{L_2}}\left\{1 - \frac{1}{q^{\frac{1}{2}}}\right\} \qquad \ldots.(24.4)$$

BOND terms $E_i$ the work index and expresses it as the amount of energy required to reduce unit mass of material from an infinite particle size to a size $L_2$ of 100 microns (i.e. $q = \infty$). The size of material is taken as the size of the square hole through which 80% of it will pass. Expressions for the work index are given in the original papers(15, 16) for various types of materials and various forms of size reduction equipment.

**Energy Utilisation**

One of the first important investigations into the distribution of the energy fed into a crusher was carried out by OWENS(7) who concluded that energy was utilised in the following ways.

1. In producing elastic deformation of the particles before fraction occurs.
2. In producing inelastic deformation which results in size reduction.
3. In causing elastic distortion of the equipment.
4. In friction between particles, and between particles and the machine.
5. In noise, heat and vibration in the plant and
6. In friction losses in the plant itself.

OWENS estimates that only about 10% of the total power is usefully employed.

During the course of an investigation by the U.S. Bureau of Mines,(8) in which a drop of weight type crusher was employed, it was found that the increase in surface was directly proportional to the input of energy but the rate of application of the load was an important factor.

This conclusion was substantiated in a more recent investigation of the power consumption in a size reduction process which is reported in three papers by KWONG and others.(10) A sample of material was crushed by putting it in a cavity in a steel mortar, placing a steel plunger over the sample and dropping a steel ball of known weight on the plunger from a measured height. Bouncing of the ball was prevented by three soft aluminium cushion wires under the mortar, and these wires were calibrated so that the energy absorbed by the system could be determined from their deformation. Losses in the plunger and ball were assumed to bear a fixed relationship to the energy absorbed by the wires, and the energy actually used for size reduction was then obtained as the difference between the energy of the ball on striking the plunger and the energy absorbed. Surfaces were measured by a water or air permeability method or by gas adsorption; the latter method gave a value approximately double that obtained from the former, indicating that, in these experiments, the internal surface was approximately the same as the external surface. The experimental results showed that, provided the new surface did not exceed about 400 cm$^2$/gm, the new surface produced was directly proportional to the energy input. For a given energy input it was independent of

(a) the velocity of impact,
(b) the weight and arrangement of the sample,
(c) the initial particle size, and
(d) the moisture content of the sample.

Between 30 and 50% of the energy of the ball on impact was absorbed by the material but no indication was obtained of how this was utilised. An extension of the range of the experiments, so that up to 1200 cm$^2$ of new surface was produced per gram of material, showed that the linear relationship between energy and new surface no longer held rigidly. However, in further experiments in which the crushing was effected slowly, using a hydraulic press, it was found that the linear relationship still held for the larger surface increases.

In order to determine the efficiency of the surface production process, experiments were carried out with sodium chloride and it was found that $900 \times 10^{-4}$ kgm.wt-cms. of energy were required to produce a square centimetre of new surface. As the theoretical value of the surface energy of sodium chloride is only $0.8 \times 10^{-4}$ kgm.wt-cms/cm², the efficiency of the process is about 0.1 %.

As indicated here, the apparent efficiency of the size reduction operation depends on the type of equipment used. Thus, for instance, a ball mill is rather less efficient than a drop weight type of crusher because of the ineffective collisions that take place.

Further work(20) on the crushing of quartz showed that more surface was created per unit of energy with single particles than with a collection of particles. This appears to be attributable to the fact that the crushing strength of apparently identical particles may vary by a factor as large as 20, and it is necessary to provide a sufficient energy concentration to crush the strongest particle.

## PARTICLE SIZE DISTRIBUTION

The variation in particle size as a result of continued grinding of a material has already been referred to, and the results of some experiments by HEYWOOD(18) have been given in Fig. 24.1. It is apparent that the product from a crusher will have a wide size distribution and that suitable methods must be developed for the purposes of representation. The size range of a material can be conveniently determined by sieving for relatively large materials, and by sedimentation methods for particles which are too fine for sieving, and the use of such methods is described in the next chapter. The results of a size analysis are generally represented by a cumulative weight fraction curve, in which the proportion of particles smaller than a certain size is plotted against the linear dimension of the particles. This curve will rise from zero to unity over the range from the size of the smallest to the largest particle, as shown in Fig. 24.2. From this curve, it is difficult to see the distribution and it is therefore useful to plot a size frequency

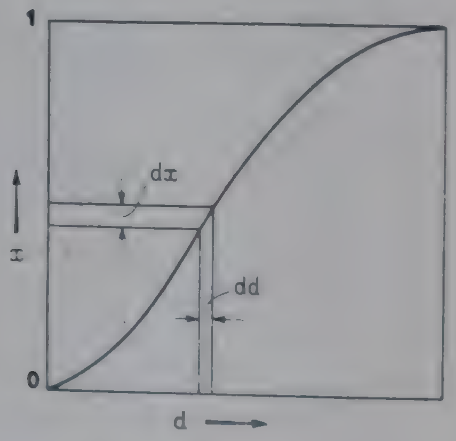

Fig. 24.2. Size distribution curve—cumulative basis

curve, which is simply the differential of the cumulative curve and is therefore obtained by plotting the slope of the curve given in Fig. 24.2. Such a curve is shown in Fig. 24.3.

The shapes of the curves obtained for the product from a size reduction system generally follow a fairly well defined form. Thus, if the cumulative curve is plotted using logarithmic co-ordinates, an approximately straight line is obtained. Again, the size frequency curve will generally show a maximum corresponding to the persistent mode in Fig. 24.1, its position being largely a function of the structure of the material. If the crusher exerts a sizing influence, for example if the material is passed through rollers with a fixed separation, there will also be a secondary maximum as shown on the right of Fig. 24.4. The frequency curve here is really formed as the sum of the two dotted curves.

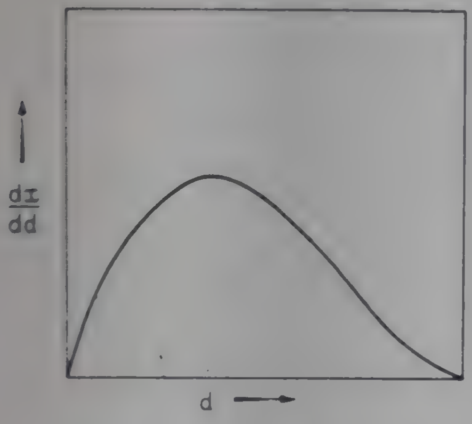

Fig. 24.3. Size distribution curve—frequency basis

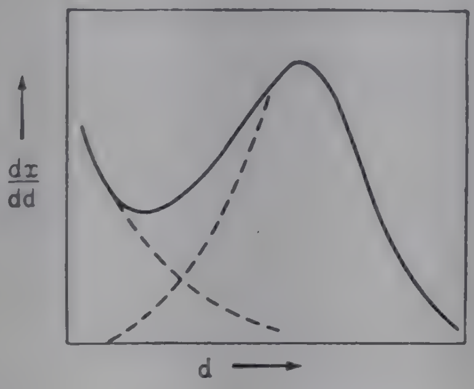

Fig. 24.4. Size frequency curve of mixed material

It is convenient to be able to express the size of a material by a single figure. Bond(15, 16) chooses the size of the opening through which 80% of the material will pass and this proves to be a good practical method of specification. It is often important however to be able to represent some particular characteristic, such as the size of the particle of mean weight or surface, and we shall now consider the various mean sizes which have been used. It should be stressed that only one property of the system is represented by this dimension, and it gives no indication of the size range. Further, it is assumed that all the particles have approximately the same shape.

**Mean Diameters Based on Volume.**

In Fig. 24.2, the area between the curve and the vertical axis is $\sum d \, . \, \mathrm{d}x$ and the mean abscissa is therefore $\dfrac{\sum d \, . \, \mathrm{d}x}{\sum \mathrm{d}x}$, or if the curve is represented by a continuous mathematical function, the abscissa is

$$\frac{\int_0^1 d \, \mathrm{d}x}{\int_0^1 \mathrm{d}x} = \int_0^1 d \, \mathrm{d}x.$$

If unit mass of mixture consists of $n_1$ particles of characteristic dimension $d_1$, constituting a mass fraction $x_1$, and $n_2$ particles of size $d_2$ etc., the mass fraction of particles of size $d_1$ is given by

$$x_1 = n_1 k_1 d_1^3 \rho_s \qquad \ldots.(24.5)$$

the total mass fraction of particles

$$= \sum x_1 = 1 = \sum n_1 k_1 d_1^3 \rho_s \qquad \ldots.(24.6)$$

and $$\sum d_1 x_1 = \sum n_1 k d_1^4 \rho_s$$

where $\rho_s$ is the density of the particles, and
$k_1$ is a constant depending on the shape of the particle.

Then the mean abscissa

$$d_v = \frac{\int_0^1 d\,\mathrm{d}x}{\int_0^1 \mathrm{d}x} = \frac{\sum d_1 x_1}{\sum x_1} = \sum d_1 x_1 \qquad \ldots.(24.7a)$$

$$= \frac{\sum n_1 d_1^4}{\sum n_1 d_1^3} \qquad \ldots.(24.7b)$$

$d_v$ is known as the volume mean diameter or as the weight mean diameter.

It will be noted that this is different from the mean volume diameter $d_v'$ which is defined as the size that each particle would have to be to make the total volume of particles the same as in the mixture.

i.e. $$k_1 d_v'^3 \sum n_1 = \sum k_1 n_1 d_1^3$$

i.e. $$d_v' = \sqrt[3]{\frac{\sum n_1 d_1^3}{\sum n_1}} \qquad \ldots.(24.8a)$$

or since $$x_1 = n_1 k_1 d_1^3 \rho_s \qquad \ldots.(24.5)$$

$$d_v' = \sqrt[3]{\frac{\sum x_1}{\sum \frac{x_1}{d_1^3}}} = \frac{1}{\sqrt[3]{\sum \frac{x_1}{d_1^3}}} \qquad \ldots.(24.8b)$$

### Mean Diameters Based on Surface

If instead of the fraction of the total mass of particles, the total surface in each mass fraction is plotted against size, we obtain a curve similar in type to that given in Fig. 24.2 and in this case, the mean abscissa is $\frac{\int d\,.\mathrm{d}S}{\int \mathrm{d}S}$ or $\frac{\sum d_1 \,.\, S_1}{\sum S_1}$

The total surface of unit mass of material

$$= \sum S_1 = \sum n_1 k_2 d_1^2$$

and $$\sum d_1 S_1 = \sum n_1 k_2 d_1^3$$

The surface mean diameter is then given by

$$d_s = \frac{\sum d_1 S_1}{\sum S_1} = \frac{\sum n_1 d_1^3}{\sum n_1 d_1^2} \qquad \ldots .(24.9a)$$

It will be noted that $d_s$ is proportional to the ratio of the total volume to total surface of the mixture, and is therefore the size of the particle with the same specific surface as the mixture. Again from equation 24.5,

$$d_s = \frac{\sum x_1}{\sum \frac{x_1}{d_1}} = \frac{1}{\sum \frac{x_1}{d_1}} \qquad \ldots .(24.9b)$$

Again it will be noted that this differs from the mean surface diameter which is given by the relation

$$k_2 d_s'^2 \sum n_1 = \sum k_2 n_1 d_1^2$$

i.e.

$$d_s' = \sqrt{\frac{\sum n_1 d_1^2}{\sum n_1}} \qquad \ldots .(24.10a)$$

and from equation 24.5

$$d_s' = \sqrt{\frac{\sum \frac{x_1}{d_1}}{\sum \frac{x_1}{d_1^3}}} \qquad \ldots .(24.10b)$$

**Linear Mean Diameters**

We also have a linear mean diameter and a mean linear diameter corresponding to those relating to volume and surface given above. The linear mean diameter $d_l$ is thus given by

$$d_l = \frac{\sum d_1 \, . \, n_1 d_1}{\sum n_1 d_1} = \frac{\sum n_1 d_1^2}{\sum n_1 d_1} \qquad \ldots .(24.11a)$$

$$= \frac{\sum \frac{x_1}{d_1}}{\sum \frac{x_1}{d_1^2}} \qquad \ldots .(24.11b)$$

The mean linear diameter is given by

$$d_l' \sum n_1 = \sum n_1 d_1$$

i.e.

$$d_l' = \frac{\sum n_1 d_1}{\sum n_1} \qquad \ldots .(24.12a)$$

and from equation 24.5,

$$d_l' = \frac{\sum \frac{x_1}{d_1^2}}{\sum \frac{x_1}{d_1^3}} \qquad \ldots .(24.12b)$$

In the above treatment, the characteristic dimension of the particle is most conveniently taken, as in Chapter 14, as the diameter of the circle of the same area as the projected area of the particle when lying in its position of maximum stability.

**Example.** The size analysis of a powdered material on a weight basis is represented by a straight line from 0% weight at 1 micron particle size to 100% weight at 101 microns particle size (Fig. 24.5). Calculate the surface mean diameter of the particles constituting the system.

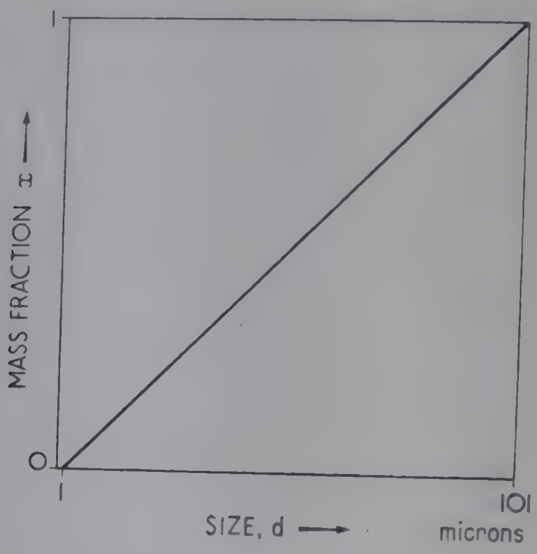

Fig. 24.5.

**Solution.** From equation 24.9*b*, the surface mean diameter is given by

$$d_s = \frac{1}{\sum \frac{x_1}{d_1}}$$

Since the size analysis is represented by the continuous curve

$$d = 100x + 1$$

$$d_s = \frac{1}{\int_0^1 \frac{dx}{d}}$$

$$= \frac{1}{\int_0^1 \frac{dx}{100x + 1}}$$

$$= \frac{100}{\ln 101}$$

$$= \underline{\underline{21.7 \text{ microns}}}$$

## METHODS OF OPERATING CRUSHERS

There are two distinct methods of feeding material to a crusher. The first is known as "free crushing" and involves feeding the material at a comparatively low rate so that the product can readily escape; its time of residence in the machine is therefore short and the production of appreciable quantities of undersize material is avoided. The second method is known as "choke feeding." In this case, the machine is kept full of material and discharge of the product is impeded so that the material remains in the crusher for a longer period. This results in a higher degree of crushing, but the capacity of the machine is reduced and energy consumption is high because of the cushioning action produced by the accumulated product. This method is therefore used only when a comparatively small amount of materials is to be crushed and when it is desired to complete the whole of the size reduction in one operation.

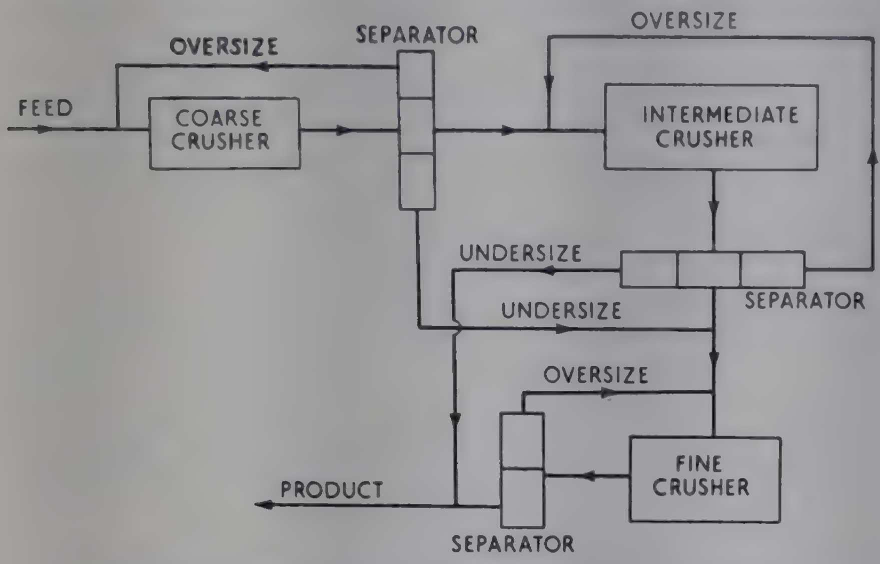

Fig. 24.6. Flow diagram for closed circuit grinding system

If the plant is operated, as in "choke feeding," so that the material is passed only once through the equipment, the process is known as "open circuit grinding." If, on the other hand, the product contains material which is insufficiently crushed, it may be necessary to separate the product and return the oversize material for a second crushing. This system is generally to be preferred and is known as "closed circuit grinding." A flow-sheet for a typical closed circuit grinding process, in which a coarse crusher, an intermediate crusher and a fine grinder are used, is shown in Fig. 24.6. In many plants, the product is continuously removed, either by allowing the material to fall on to a screen or by subjecting it to the action of a stream of fluid: the small particles are carried away and the oversize material falls back to be crushed again.

The desirability of using a number of size reduction units when the particle size is to be considerably reduced arises from the fact that it is not generally economical to effect a large reduction ratio in a single machine. The equipment is usually roughly divided into classes as given below according to the size of the feed and the product

| | *Feed Size* | *Product Size* |
|---|---|---|
| Coarse crushers | 60″–1½″ (150–4 cm) | 2″–¼″ (5–0·5 cm) |
| Intermediate crushers | 2″–¼″ (5–0·5 cm) | ¼″–200 mesh (0·5–0·01 cm) |
| Fine crushers | ¼″–⅛″ (0·5–0·2 cm) | about 200 mesh (0·01 cm) |
| Colloid mills | 80 mesh (0·02 cm) | down to $10^{-6}$″ (0·01 microns) |

A greater size reduction ratio can be obtained in fine crushers than in coarse crushers.

The equipment can also be classified, to some extent, according to the nature of the force which is applied but, as generally a number of forces are involved, it is a less convenient basis.

Grinding can be carried out either wet or dry but wet grinding is generally applicable only with low speed mills. The advantages of wet grinding are

1. The power consumption is less by about 20–30%.
2. The capacity of the plant is increased.
3. Removal of the product is facilitated and the amount of fines is reduced.
4. Dust formation is eliminated.
5. The solids are more easily handled.

Against this, the wear on the grinding medium will generally be about 20% greater, and it may be necessary to dry the product.

## NATURE OF THE MATERIAL TO BE CRUSHED

The choice of machine for a given crushing operation will be affected by the nature of the product required and the quantity and size of material to be handled. The more important properties of the feed apart from its size are as follows:

*Hardness*. The hardness of the material affects the power consumption and the wear on the machine. With hard and abrasive materials it is necessary to use a low-speed machine and to protect the bearings from the abrasive dusts that are produced. Pressure lubrication is recommended. Materials are arranged in order of increasing hardness in the Mohr scale. The first four items rank as soft and the remainder as hard.

*Mohr Scale of Hardness*

1. Talc
2. Rock salt or gypsum
3. Calcite
4. Fluorspar
5. Apatite
6. Felspar
7. Quartz
8. Topaz
9. Carborundum
10. Diamond

*Structure*. Normal granular materials such as coal, ores and rocks can be effectively crushed employing the normal forces of compression, impact, etc. With fibrous materials it is necessary to effect a tearing action.

*Moisture Content*. It is found that materials do not flow well if they contain between about 5 and 50% of moisture; under these conditions the material tends to cake together in the form of balls. Grinding can be carried out satisfactorily, in general, outside these limits.

*Crushing Strength*. The power required for crushing is almost directly proportional to the crushing strength of the material.

*Friability*. The friability of the material is its tendency to fracture during normal handling. In general, a crystalline material will break along well defined planes and the power required for crushing will increase as the particle size is reduced.

*Stickiness.* A sticky material will tend to clog the grinding equipment and therefore it should be ground in a plant that can be readily cleaned.

*Soapiness.* This is, in general, a measure of the coefficient of friction of the surface of the material. If the coefficient of friction is low the crushing may be more difficult.

*Explosive Materials* must be ground wet or in the presence of an inert atmosphere.

*Materials yielding dusts that are harmful to the health* must be ground under conditions where the dust is not allowed to escape.

## TYPES OF CRUSHING EQUIPMENT

The most important coarse, intermediate and fine crushers are as follows.

| *Coarse Crushers* | *Intermediate Crushers* | *Fine Crushers* |
|---|---|---|
| Blake Jaw Crusher | Crushing Rolls | Buhrstone Mill |
| Dodge Jaw Crusher | Disc Crusher | Roller Mill |
| Gyratory Crusher | Edge Runner Mill | Raymond Mill |
| Samson Crusher | Conical Crusher | Griffin Mill |
| | Stamp Battery | Centrifugal Ball Mill |
| | Hammer Mill | Ring Roller Mill |
| | Single Roll Crusher | Ball Mill |
| | Pin Mill | Tube Mill |
| | End Runner Mill | Hardinge Mill |
| | Squirrel Cage Disintegrator | |

## COARSE CRUSHERS

### The Blake Jaw Crusher

The Blake jaw crusher (Fig. 24.7) has a fixed jaw and a moving jaw pivoted at the top. The crushing faces themselves are formed either of manganese steel or of chilled cast iron and must be carefully fitted because they are brittle; the risk of breakage is reduced by grinding the back surface flat or packing with lead. Since the maximum movement of the jaw is at the bottom, there will be little tendency for the machine to clog though some uncrushed material may fall through and have to be returned to the crusher. Further, the maximum pressure will be exerted on the large material which is introduced at the top. The machine is usually protected so that it is not damaged if lumps of metal inadvertently enter, by making one of the toggle plates in the driving mechanism relatively weak so that, if any large stresses are set up, this is the first part to fail. Easy renewal of the damaged part is then possible.

Blake crushers are made with jaw widths varying from about 2 in. to 48 in. and the running speed varies from about 100 to 400 r.p.m., the smaller machines running at the higher speeds. The speed of operation should not be so high that a large quantity of fines is produced as a result of material being repeatedly crushed because it cannot escape sufficiently quickly. The angle of nip, the angle between the jaws, is usually about 30°. Because the crushing action is intermittent, the loading on the machine is uneven and the crusher therefore incorporates a heavy flywheel.

The Samson crusher (Fig. 24.8) is similar to the Blake, but the movable jaw is pivoted from the same axis as the drive. Its characteristics are similar.

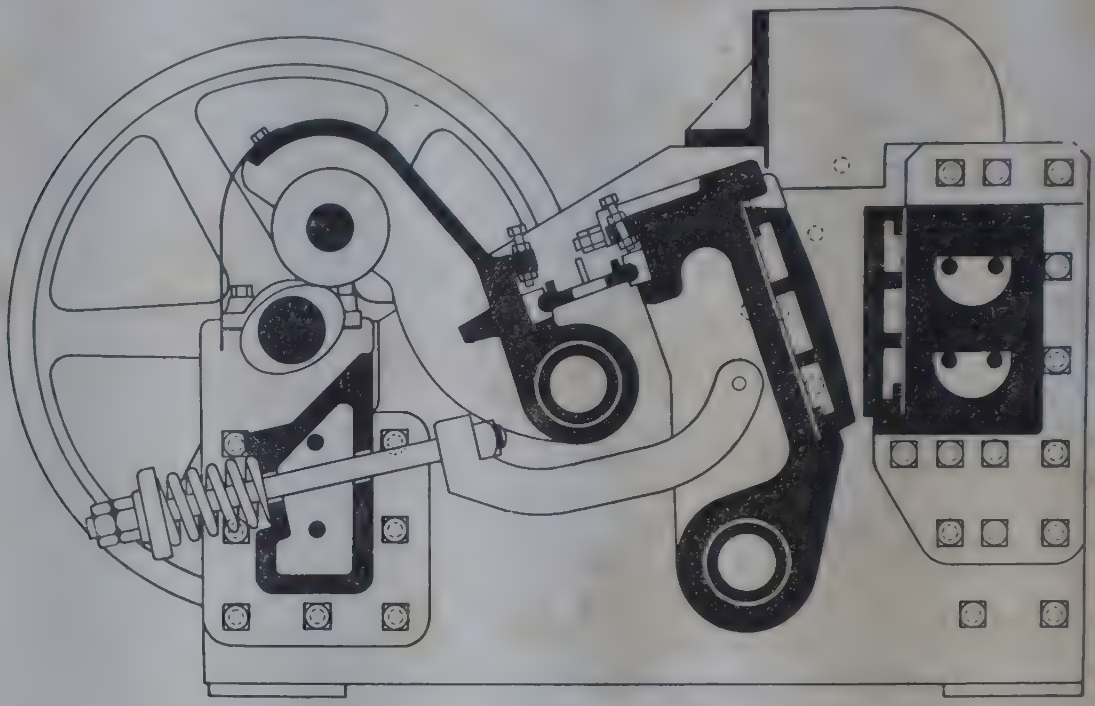

Fig. 24.9. Dodge crusher

### The Dodge Jaw Crusher

In the Dodge crusher (Fig. 24.9) the moving jaw is pivoted at the bottom. The minimum movement is thus at the bottom and a more uniform product is obtained, but the crusher is less widely used because of its tendency to choke. The large opening at the top enables it to take very large feed and to effect a large size reduction. This crusher is usually made in smaller sizes than the Blake crusher, because of the high fluctuating stresses that are produced in the members of the machine.

### The Gyratory Crusher

The gyratory crusher (Fig. 24.10) employs a crushing head, in the form of a truncated cone, mounted on a shaft, the upper end of which is held in a flexible bearing, whilst the lower end is driven eccentrically so as to describe a circle. The crushing action takes place round the whole of the cone and, since the maximum movement is at the bottom, the characteristics of the machine are similar to those of the Blake crusher. As the crusher is continuous in action, the fluctuations in the stresses are smaller than in the jaw crushers and the power consumption is lower. It has a large capacity per unit area of grinding surface, particularly if it is used to produce a small size reduction. However, it

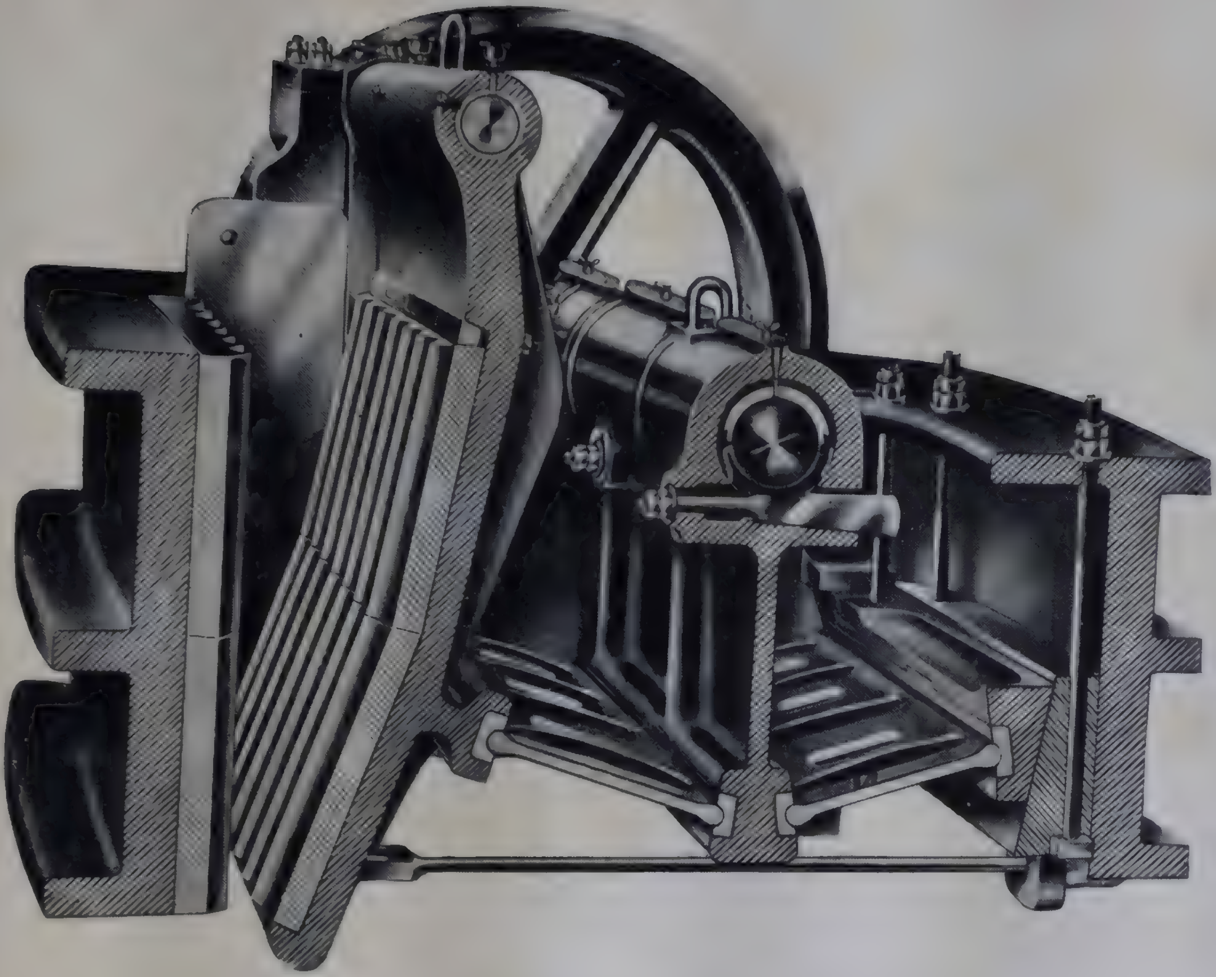

Fig. 24.7. Blake jaw crusher

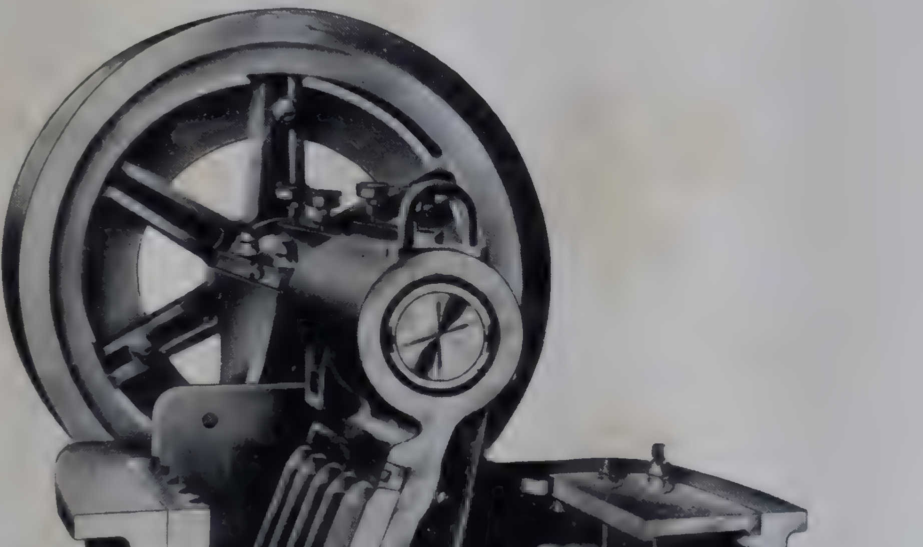

Fig. 24.8. Samson crusher—single toggle machine

will not take such large feed as a jaw crusher with the same throughput but it will give a rather finer and more uniform product. Because the capital cost is high, the crusher is suitable only where large quantities of material are to be handled.

The jaw crushers and the gyratory crusher both employ a predominantly compressive force.

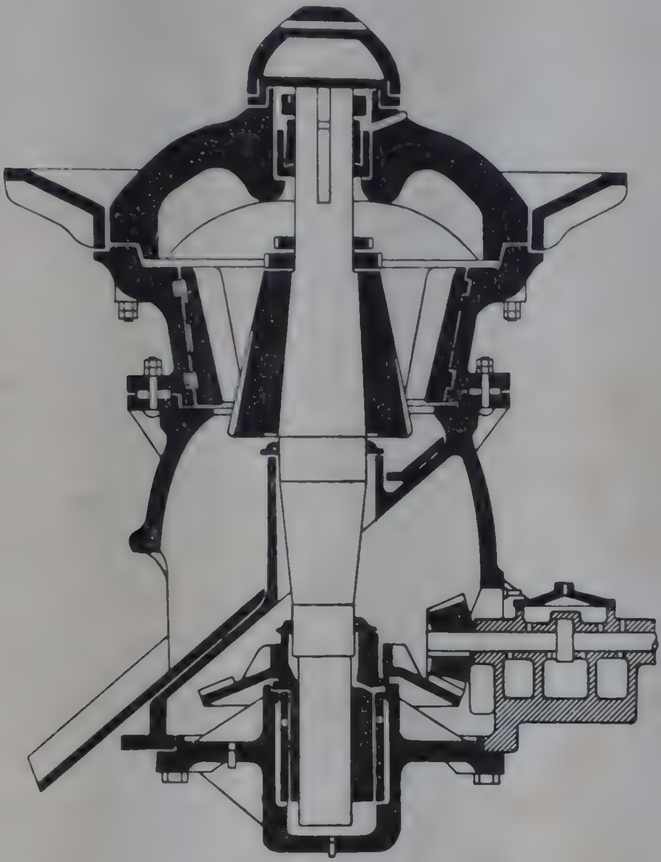

Fig. 24.10. Gyratory crusher

### Other Coarse Crushers

Friable materials, such as coal, can be broken up without the application of large forces and therefore less robust plant can be used. A common form of coal breaker consists of a large hollow cylinder with perforated walls. The axis is at a small angle to the horizontal and the feed is introduced at the top. The cylinder is rotated and the coal is lifted by means of arms attached to the inner surface and then falls against the cylindrical surface. The coal breaks by impact and passes through the perforations as soon as the size has been sufficiently reduced. This type of equipment is less expensive and has a higher throughput than the jaw or gyratory crushers. Another coarse rotary breaker, similar in action to the hammer mill, is shown in Fig. 24.11.

## INTERMEDIATE CRUSHERS

### The Stamp Battery

The stamp battery (Fig. 24.12) has been used extensively for moderately fine crushing in the past but it has now been very largely superseded by more efficient equipment. It consists of a number of heavy stamps—up to 1000 to

2000 pounds in weight—which are raised mechanically and allowed to fall under gravity on to the material to be crushed. The larger the number of stamps in

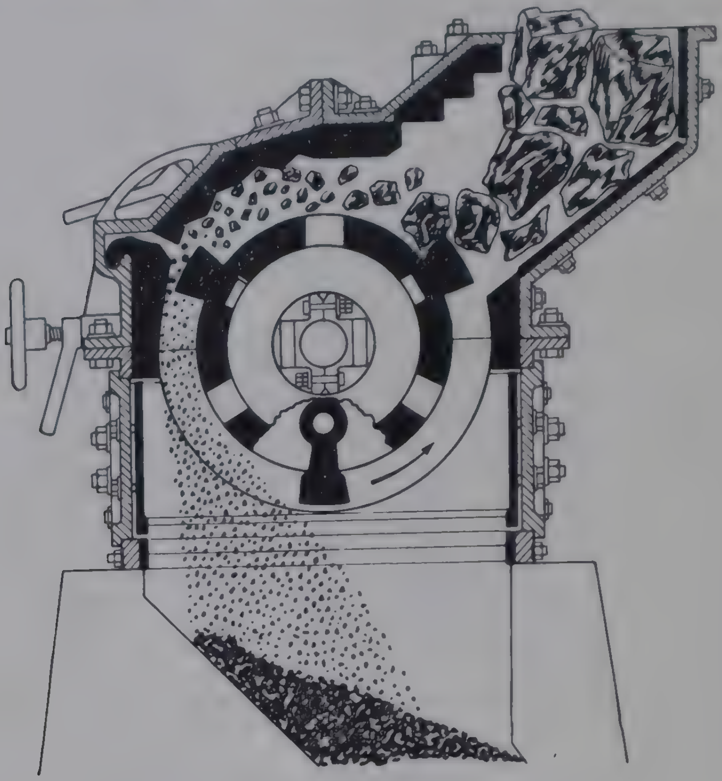

Fig. 24.11. Rotary coal breaker

the battery, the more even is the load on the driving mechanism. The solids are usually fed in as a suspension in water so that the product can be continuously removed from the system.

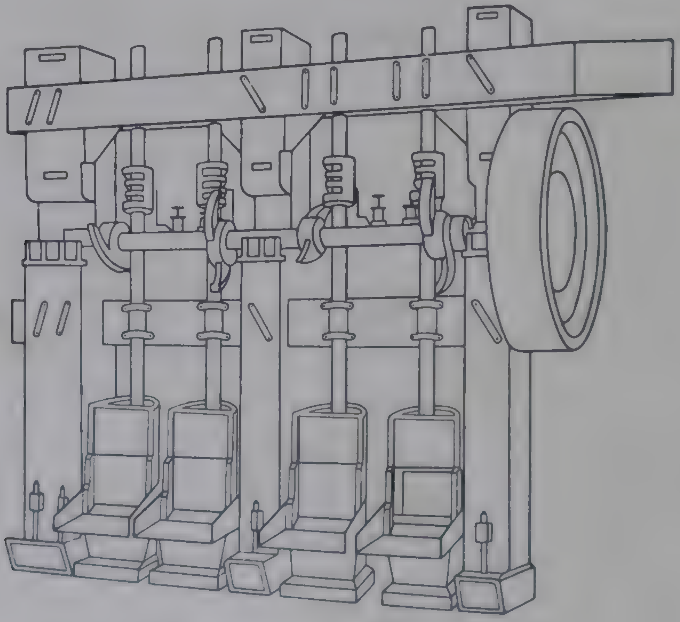

Fig. 24.12. Stamp battery

### The Edge Runner Mill

In the edge runner mill (Fig. 24.20) a heavy cast iron or granite wheel, or muller as it is called, is mounted on a horizontal shaft which is rotated in a horizontal plane in a heavy pan; alternatively, the muller remains stationary and the pan is rotated. In some cases the mill incorporates two mullers. Material is fed to the centre of the pan and is worked outwards by the action of the muller whilst a scraper continuously removes material that has adhered to the sides of the pan, and returns it to the crushing zone. In many models the outer rim of the bottom of the pan is perforated, so that the product can be removed continuously as soon as its size has been sufficiently reduced. The mill can be operated wet or dry and is used extensively for the grinding of paints, clays and sticky materials.

### The End Runner Mill

The end runner mill (Fig. 24.21) is usually made in small laboratory sizes and consists of a cast iron or porcelain mortar which is rotated so that grinding takes place against a cylindrical pestle mounted with its axis vertical. Material is continuously scraped from the sides of the mortar with a doctor knife. A fine product can usually be obtained.

### The Hammer Mill

The hammer mill is an impact mill employing a high speed rotating disc, to which are fixed a number of hammer bars which are swung outwards by centrifugal force. Two industrial models are illustrated in Figs. 24.13 and 24.14 and a laboratory model in Fig. 24.22. The material is fed in, either at the top or at the centre, and is thrown out centrifugally and crushed by being beaten between the hammer bars, or against breaker plates fixed around the periphery of the cylindrical casing. The material is beaten until it is small enough to fall through the screen which forms the lower portion of the casing. Since the hammer bars are hinged the presence of any hard material does not cause damage to the equipment. The bars are readily replaced when they are worn out. The machine

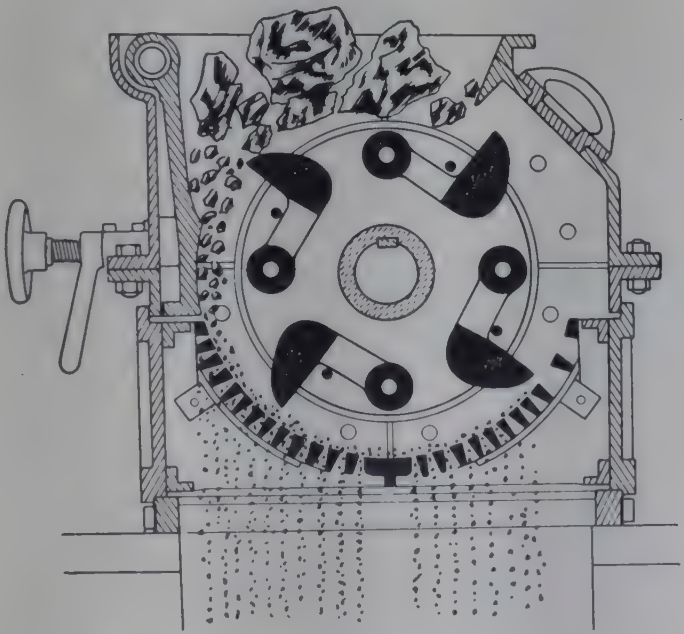

Fig. 24.13. Swing claw hammer mill

is suitable for the crushing of both brittle and fibrous materials and, in the latter case, it is usual to employ a screen with cutting edges. The hammer mill is suitable for hard materials but since a large amount of fines is produced, it is advisable to employ positive pressure lubrication to the bearings in order to prevent the entry of dust. The size of the product is regulated by the size of the screen and the speed of rotation.

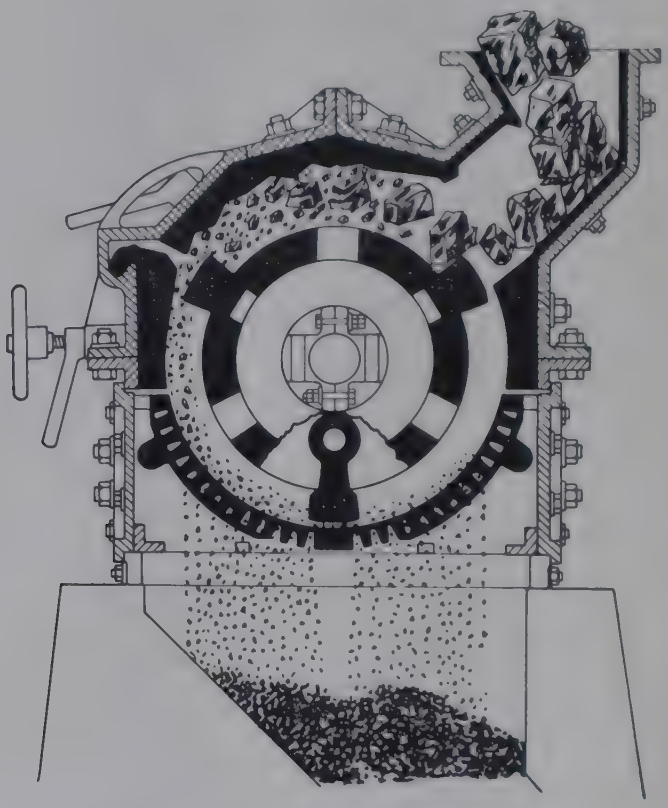

Fig. 24.14. Swing hammer mill

A number of similar machines are made, and in some the hammer bars are rigidly fixed in position. Since a large current of air is produced, the dust must be separated in a cyclone separator or a bag filter.

### The Pin Type Mill

The Kek mill (Fig. 24.23) is a form of pin mill and consists of two horizontal steel plates with vertical projections on their near faces. The upper disc is stationary whilst the lower disc is rotated at high speed. The material is fed in through a hopper in the centre of the upper disc, and is thrown outwards by centrifugal action and broken against the projections. The mill gives a fairly uniform fine product with little dust and is extensively used with chemicals, fertilisers and other materials that are non-abrasive and easily broken. Control of the size of the product is effected by means of the speed and the spacing of the projections.

The squirrel cage disintegrator or bar mill (Fig. 24.24) is similar in action but employs vertical discs with horizontal bars. It is used with friable materials, such as coal and limestone, and with fibrous materials.

## The Single Roll Crusher

The single roll crusher (Fig. 24.15) consists of a toothed crushing roll which rotates close to a breaker plate. The material is crushed by compression and shearing between the two surfaces. It is used extensively in crushing coal and in the model shown in Fig. 24.25, the coal is crushed in three stages.

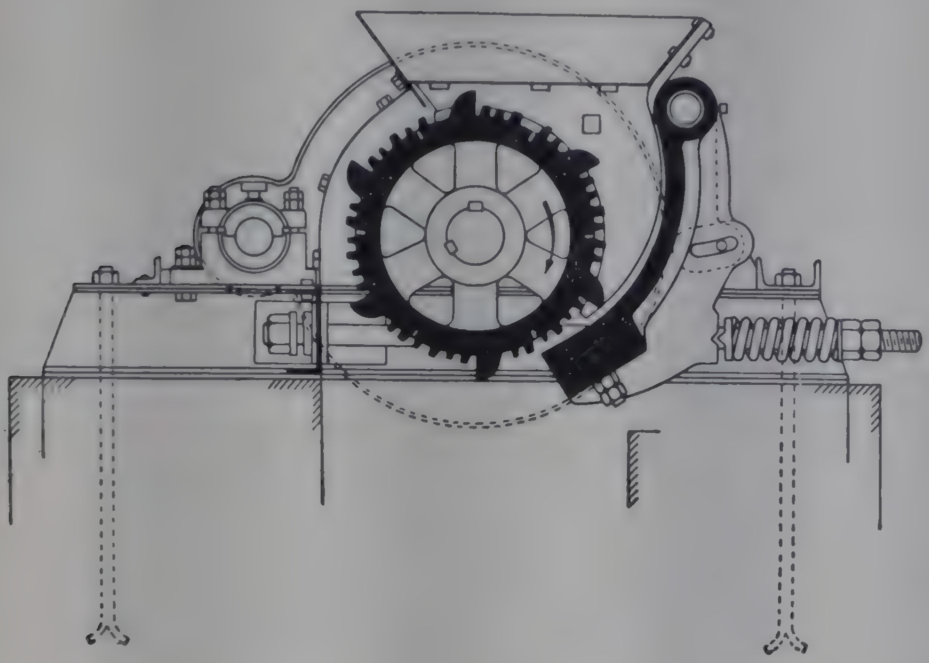

Fig. 24.15. Single roll crusher

## Crushing Rolls

Two rolls, one in adjustable bearings, rotate in opposite directions and the clearance between them can be adjusted according to the size of feed and the required size of product (Figs. 24.16 and 24.26). The machine is protected, by spring loading, against damage from very hard material. Generally, one roll is driven directly and the other by friction with the solids. The crushing rolls, which may be from a few inches up to about 4 feet in diameter, are suitable for effecting only a small size reduction in a single operation and it is therefore

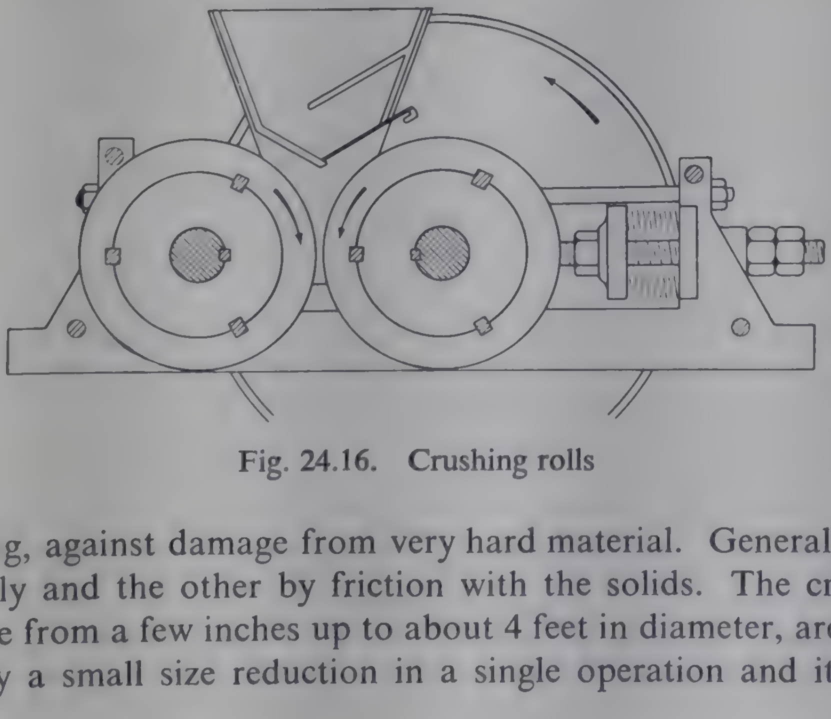

Fig. 24.16. Crushing rolls

common to employ a number of pairs of rolls in series, one above the other. Tyres with either smooth or ridged surfaces are keyed to the rolls. The capacity is usually between $\frac{1}{10}$ and $\frac{1}{3}$ of that calculated on the assumption that a continuous ribbon of the material forms between the rolls.

Fig. 24.17 shows an idealised system where a spherical or cylindrical particle of radius $r_2$ is being fed to crushing rolls of radius $r_1$. $2\alpha$ is the angle of nip, the angle between the two common tangents to the particle and each of the rolls, and $2b$ is the distance between the rolls. It will be seen from the geometry of the system that the angle of nip is given by

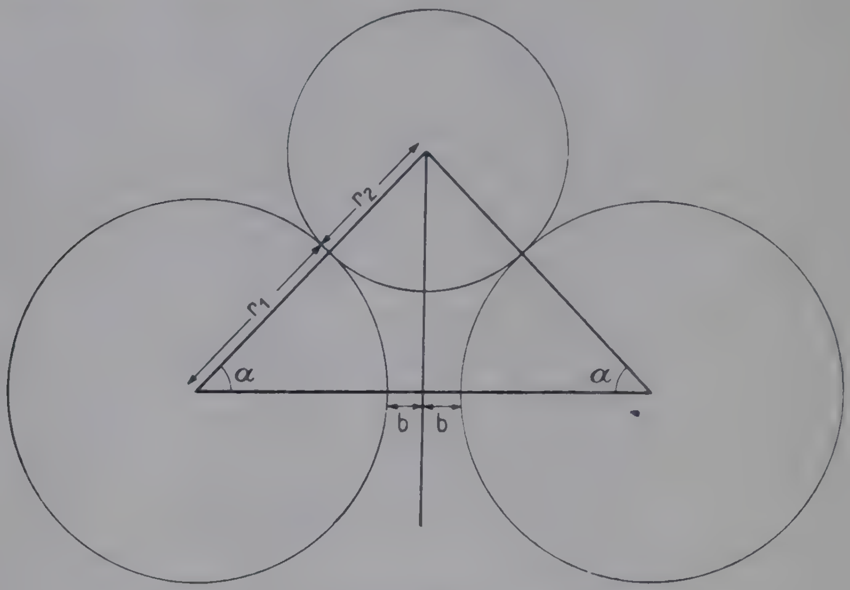

Fig. 24.17. Particle fed to crushing rolls

$$\frac{r_1 + b}{r_2 + r_1} = \cos \alpha \qquad \ldots .(24.13)$$

For steel rolls, the angle of nip will be not less than about 32°.

Crushing rolls are extensively used in crushing oil seeds and in the gunpowder industry and are also suitable for abrasive materials. They are simple in construction and do not give a large percentage of fines.

**Conical Crushers**

Conical crushers are now replacing many of the other types of intermediate crusher. They are similar in construction to the gyratory crusher, though they will not take such a coarse feed and give a very much finer product; they operate at rather higher speeds. In some conical grinders (Fig. 24.27), the crushing head is given a rotating motion instead of an eccentric motion. For efficient operation, the conical grinders should be supplied with dry and uniformly sized material and are therefore most suited to closed circuit grinding; they give free discharge of product.

### The Symons Disc Crusher

The disc crusher (Fig. 24.18) employs two saucer shaped discs mounted on horizontal shafts, one of which is rotated and the other is mounted in an eccentric bearing so that the two crushing faces continuously approach and recede. The material is fed into the centre between the two discs and the product is discharged by centrifugal action as soon as it is fine enough to escape through the opening between the faces.

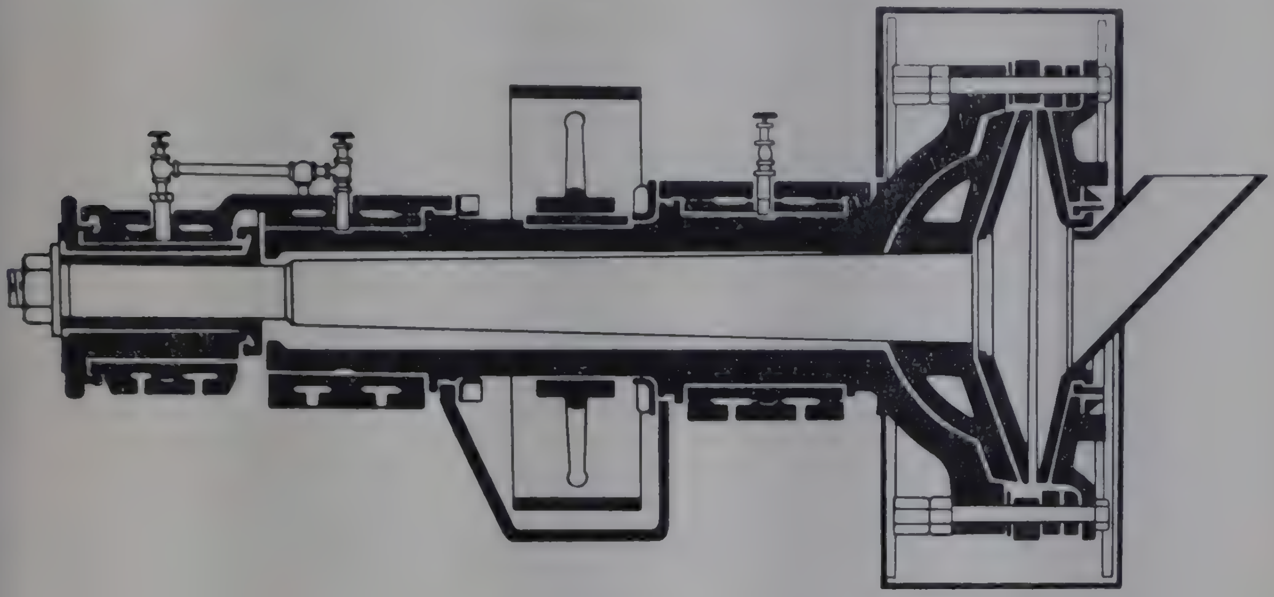

Fig. 24.18. Symons disc crusher

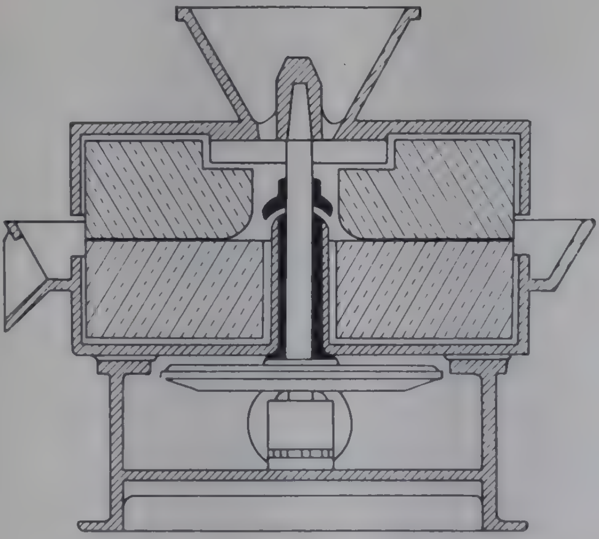

Fig. 24.19. Buhrstone mill

## FINE CRUSHERS

### The Buhrstone Mill

The Buhrstone mill (Fig. 24.19) is one of the oldest forms of fine crushing equipment though it has, very largely, been superseded now by roller mills. Grinding takes place between two heavy horizontal wheels, one of which is stationary and the other driven. The surface of the stones is carefully dressed so that the material is continuously worked from the centre to the circumference of the wheels. Size reduction takes place by a shearing action between the edges of the grooves on the two grinding stones. This equipment has been used in the past for the grinding of grain, pigments for paints, pharmaceuticals, cosmetics and printers' ink but is now used only where the quantity of material is very small.

**Roller Mills**

The roller mill consists of a pair of rollers that rotate at different speeds (e.g. 3 : 1 ratio) in opposite directions. As in crushing rolls, one of the rollers is held in a fixed bearing whereas the other has an adjustable spring loaded bearing. Since the rollers rotate at different speeds, size reduction is effected by a combination of compressive and shear forces. The roller mill is now extensively used in the flour milling industry and for the manufacture of pigments for paints.

**Centrifugal Attrition Mills**

***The Babcock Mill.*** This mill (Fig. 24.28), like the Lopulco and Raymond mills, employs centrifugal force for size reduction. It consists of a series of pushers which cause heavy cast iron balls to rotate against a bull ring like a ball race, and the pressure of the balls on the bull ring is produced entirely by centrifugal action. Material fed into the mill falls on the bull ring, and the product is continuously removed in an upward stream of air which carries the ground material between the rotating blades of the classifier, which is shown towards the top of the photograph; the oversize material falls back and is reground. The air stream is produced by means of an external blower and may involve a considerable additional power consumption. The fineness of the product is controlled by the rate of feeding and the air velocity. This machine is used mainly for preparation of pulverised coal and sometimes for cement.

***The Lopulco Mill or Ring Roll Pulveriser.*** These machines (Fig. 24.29) are being manufactured in large numbers at the present time for the production of pulverised coal at power stations. A slightly concave circular bull ring rotates at high speed and the feed is thrown outwards by centrifugal action under the crushing rollers, which are shaped like truncated cones (Fig. 24.30). The rollers are spring-loaded and the strength of the springs determines the crushing force available. There is always a clearance between the rollers and the bull ring so that there is no wear on the grinding heads if the plant is operated light, and quiet operation is obtained. The product is continuously removed by means of a stream of air produced by an external blower and is carried into a separator fitted above the grinding mechanism. In this separator, the cross-sectional area for flow is gradually increased and, as the air velocity falls, the oversize material falls back and is ground again. The product is separated in a cyclone separator from the air which is then recycled. Chemicals, dyes, cements, and phosphate rocks are also ground in the Lopulco mill. As the risk of sparking is reduced by the maintenance of a clearance between the grinding media, the mill can be used for grinding explosive materials.

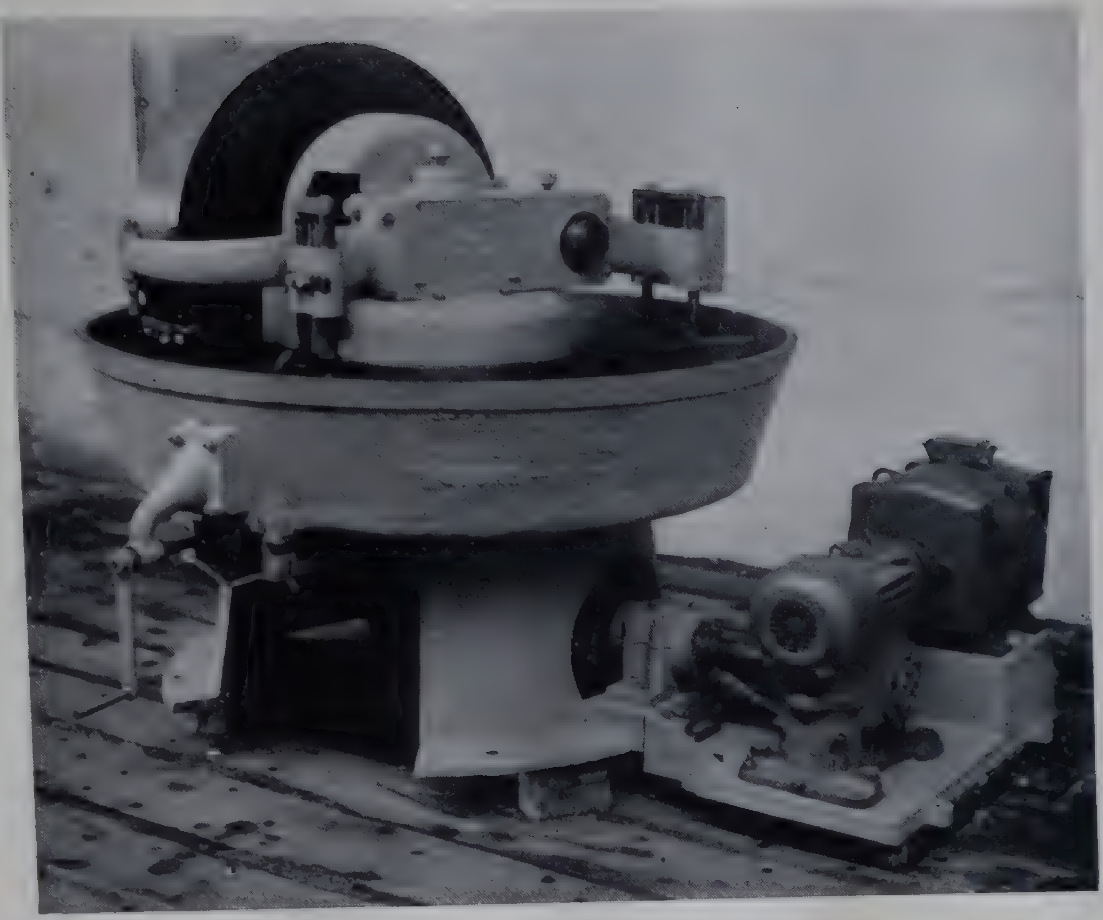

Fig. 24.20. Edge runner mill

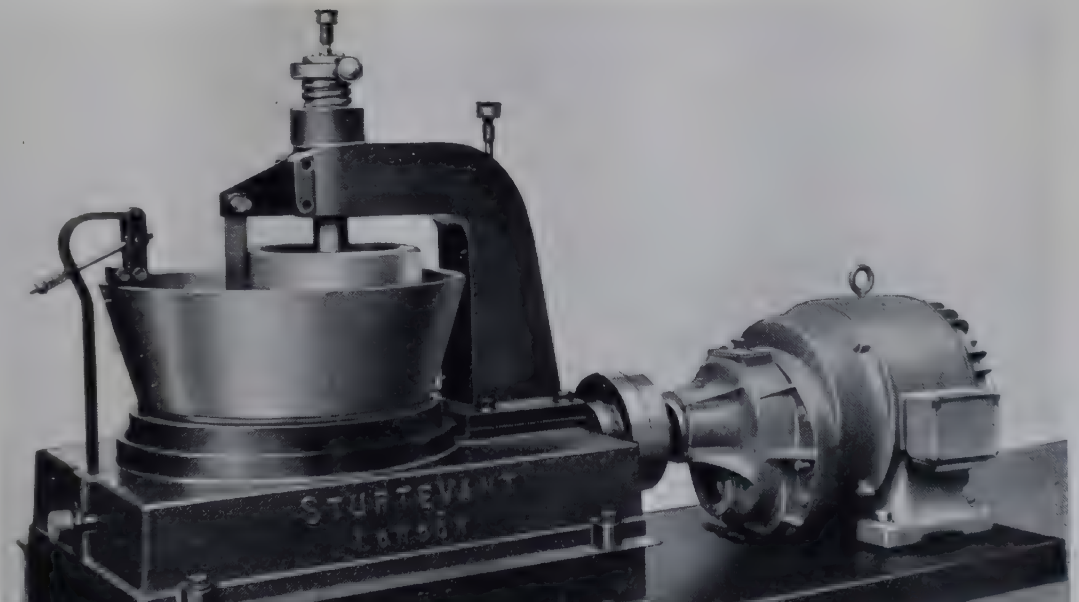
STURTEVANT
LONDON

Fig. 24.21. End runner mill

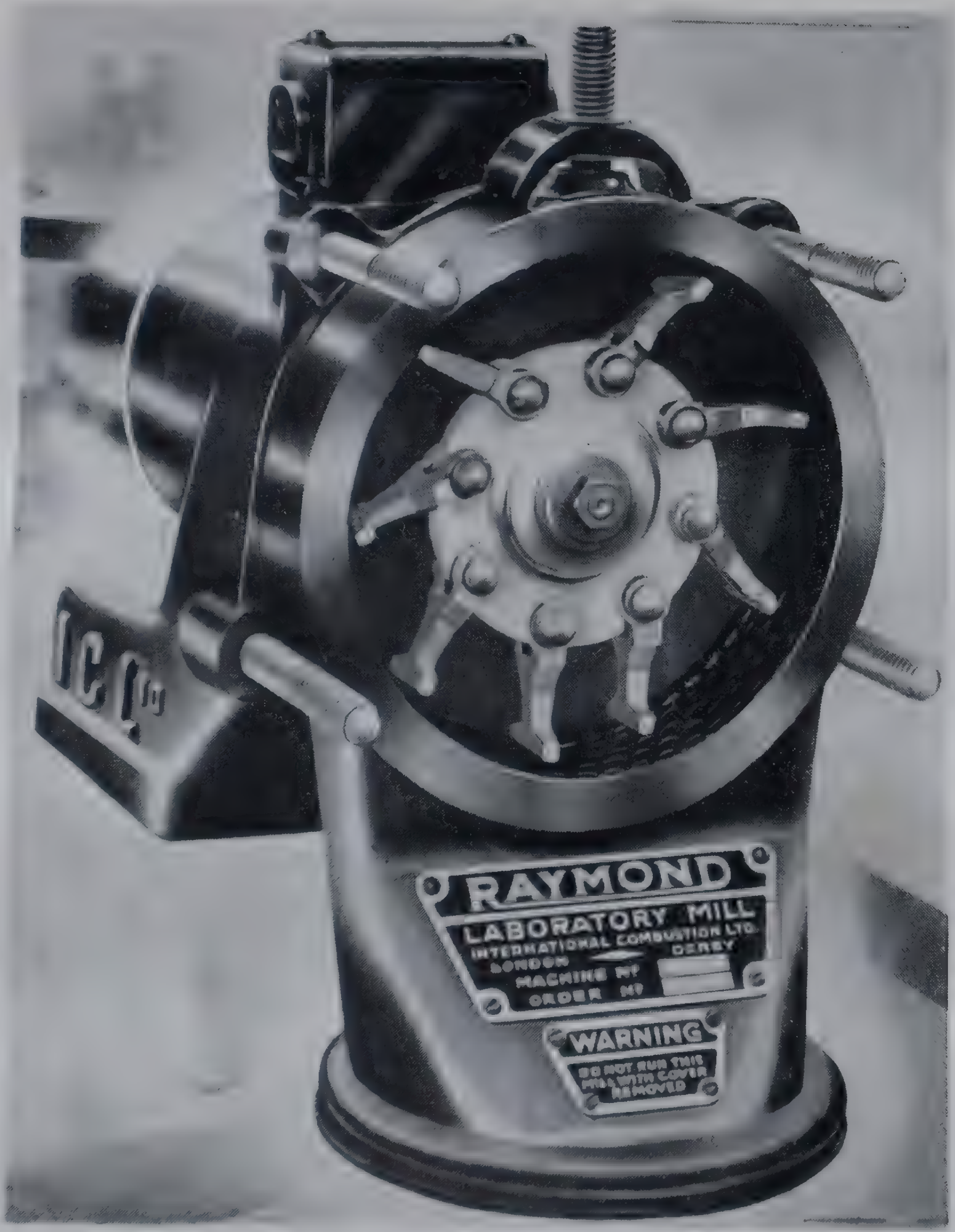
RAYMOND
LABORATORY MILL
INTERNATIONAL COMBUSTION LTD.
LONDON DERBY
MACHINE Nº
ORDER Nº
WARNING
DO NOT RUN THIS MILL WITH COVER REMOVED

Fig. 24.22. Laboratory hammer mill

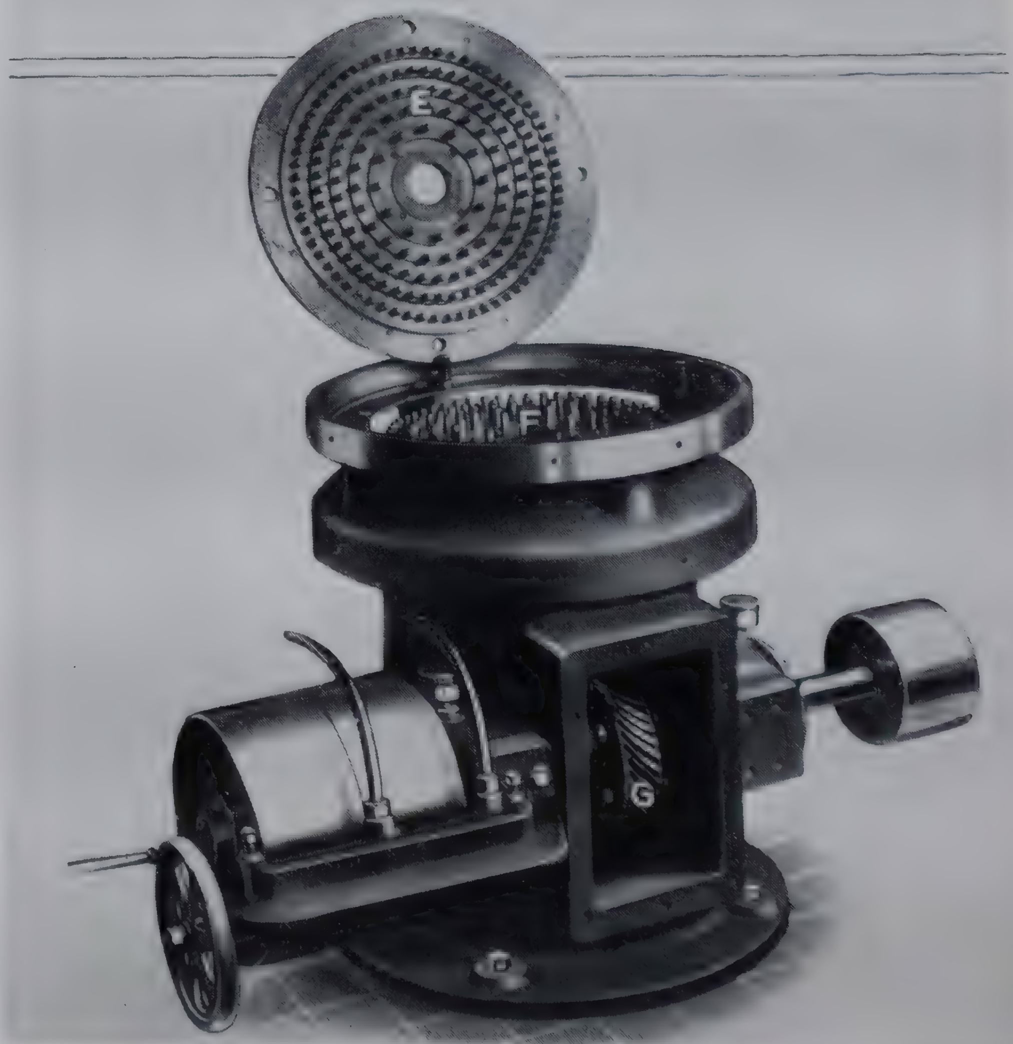

Fig. 24.23. Kek mill

*E*—Fixed pins *F*—Rotating pins *G*—Drive

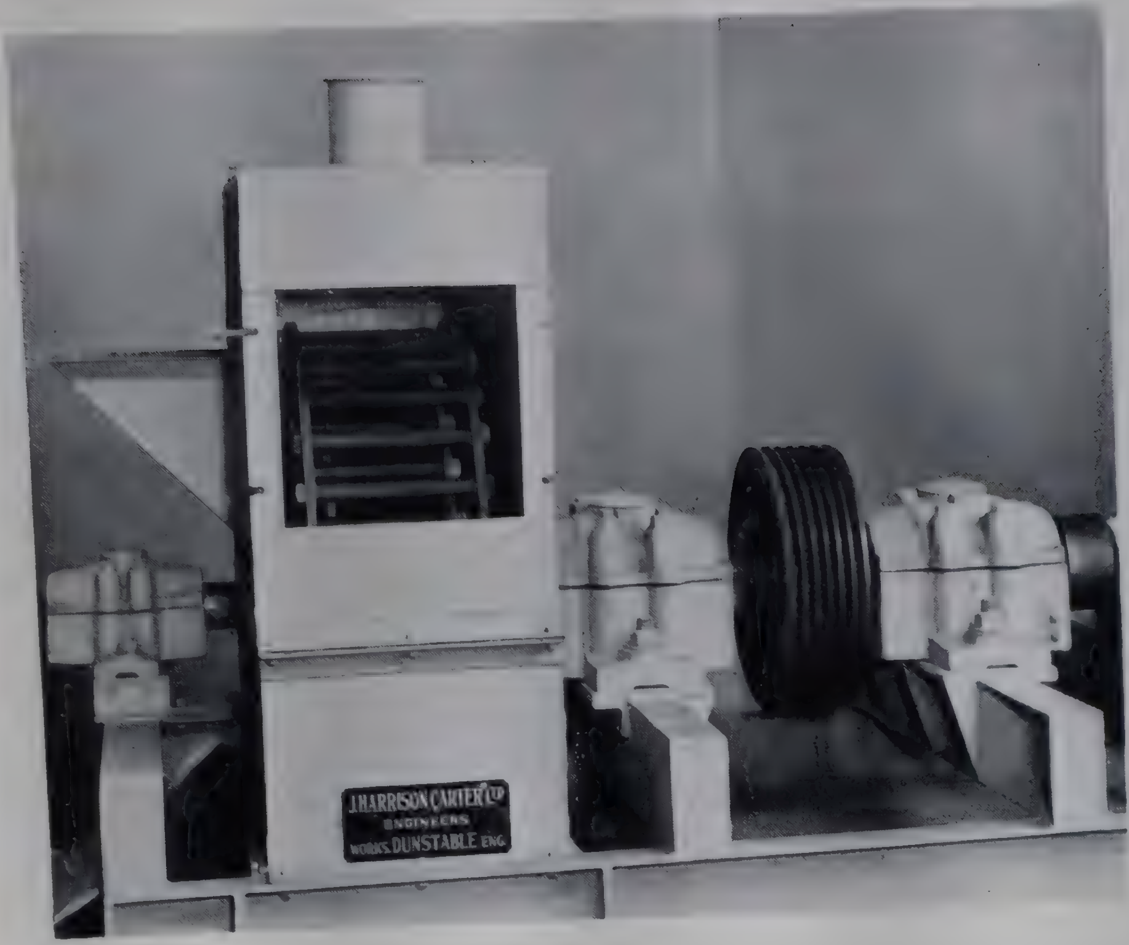
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Fig. 24.24. Bar mill

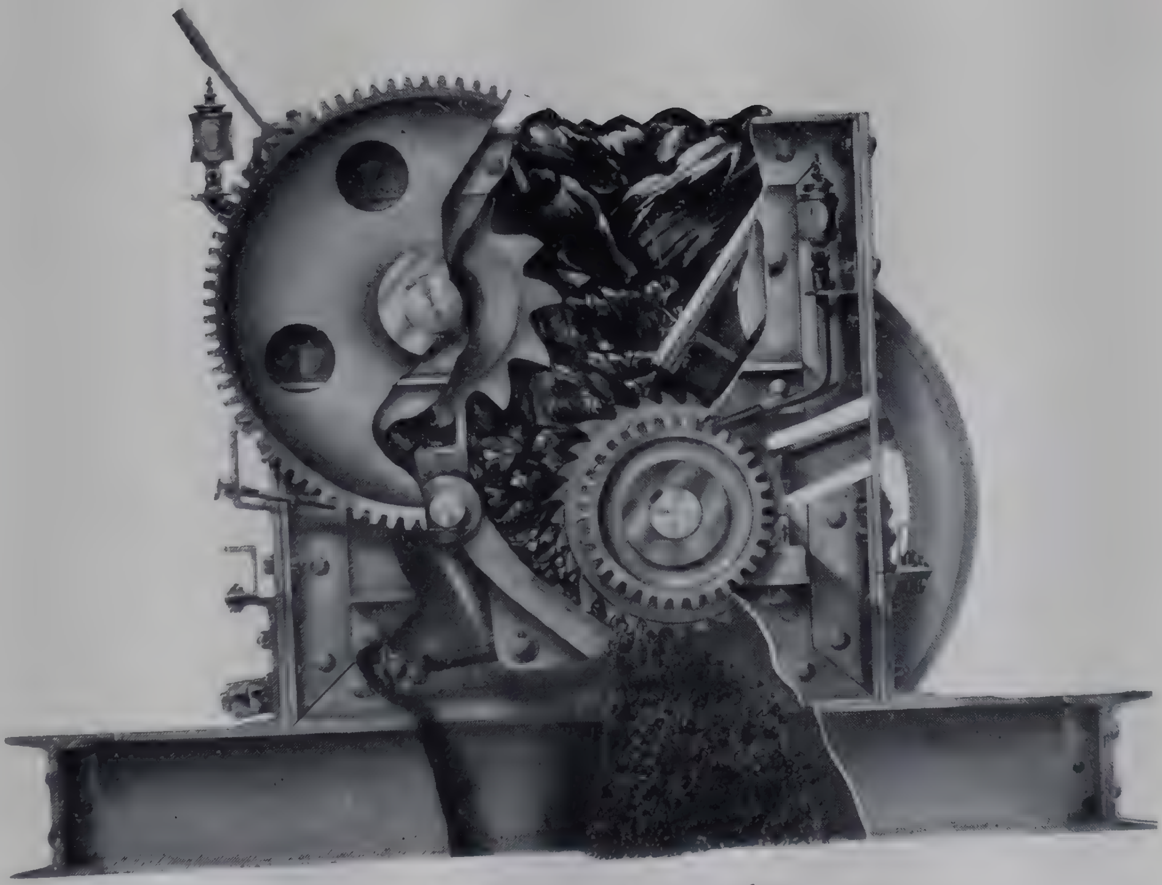


Fig. 24.25. Three stage breaker

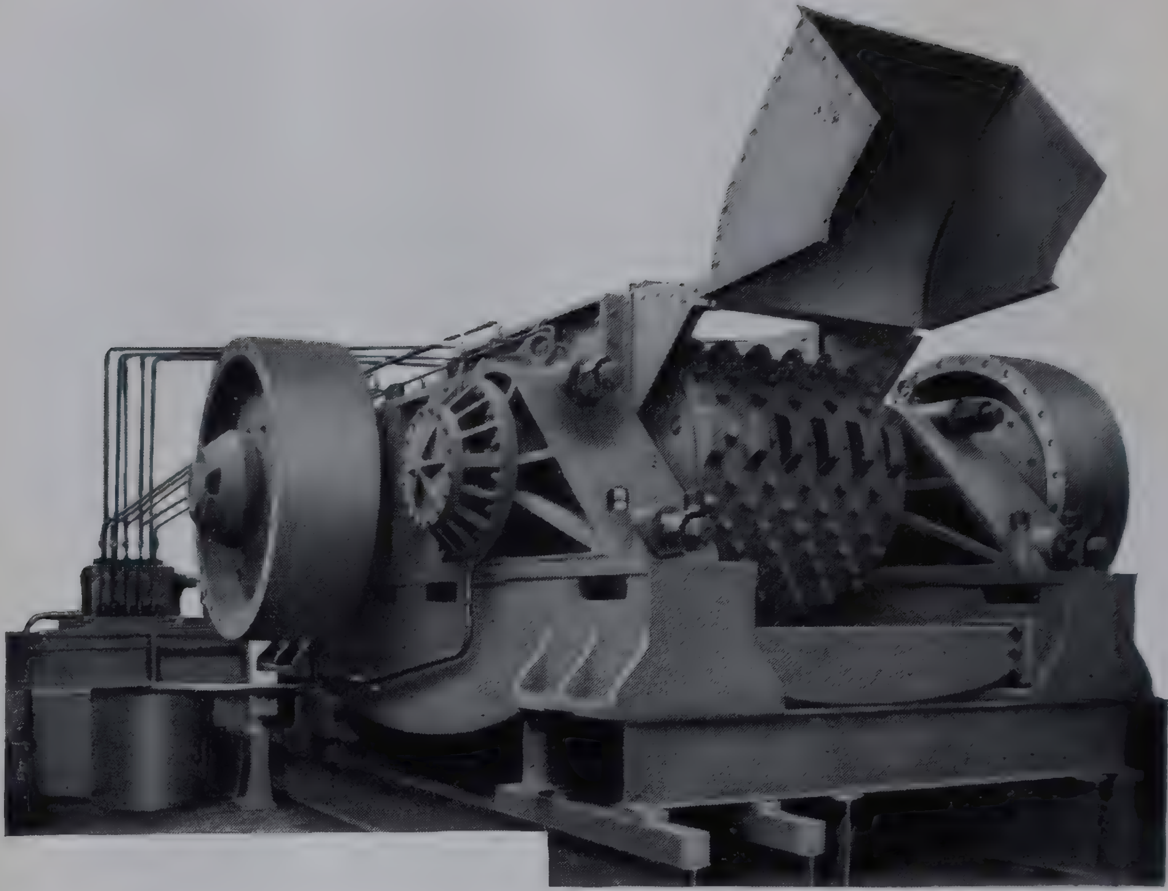


Fig. 24.26. Crushing rolls

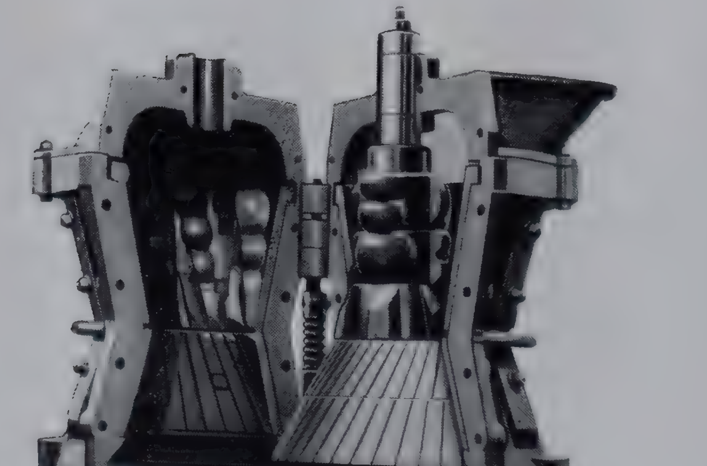


Fig. 24.27. Conical grinder

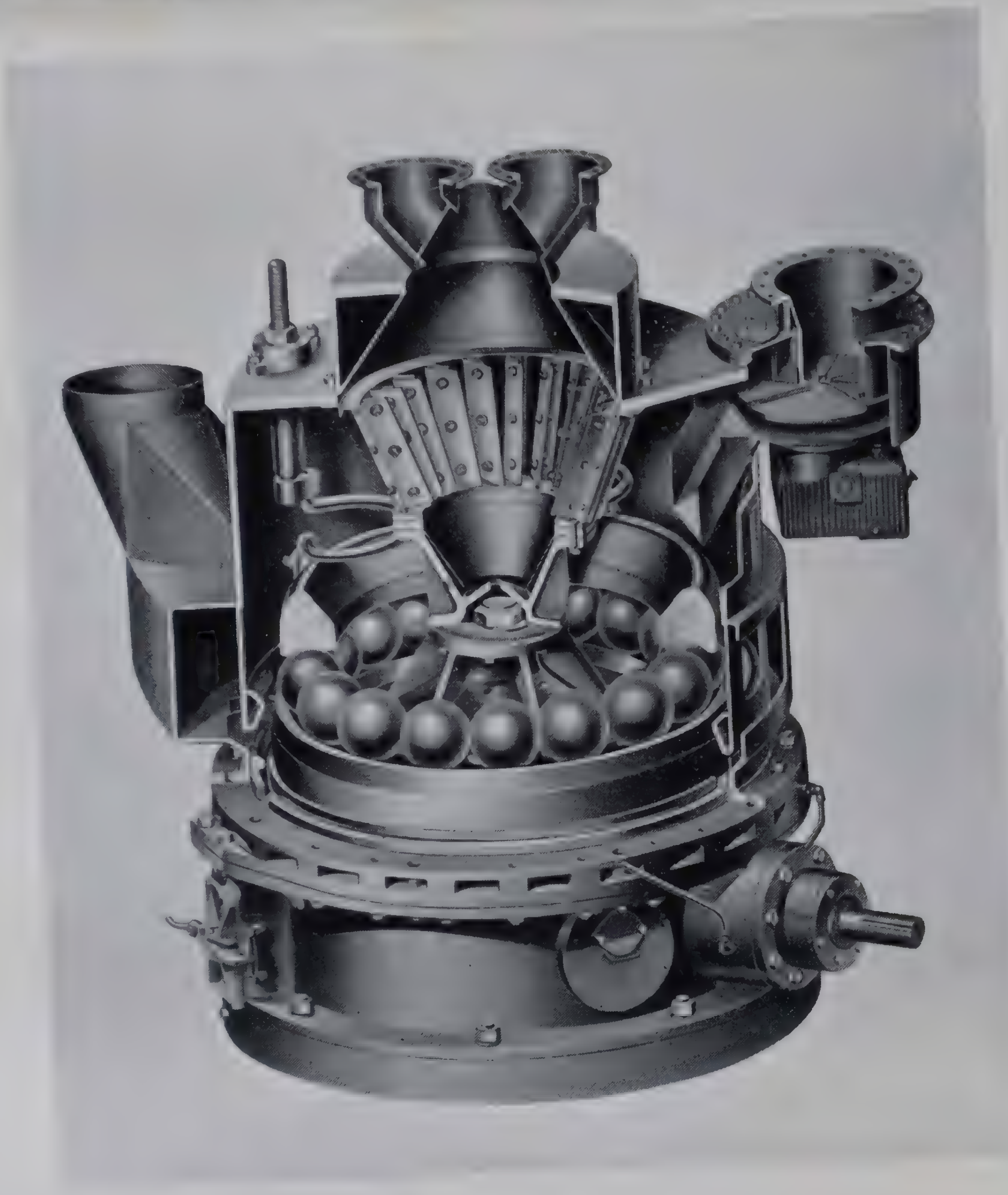


Fig. 24.28. Babcock mill

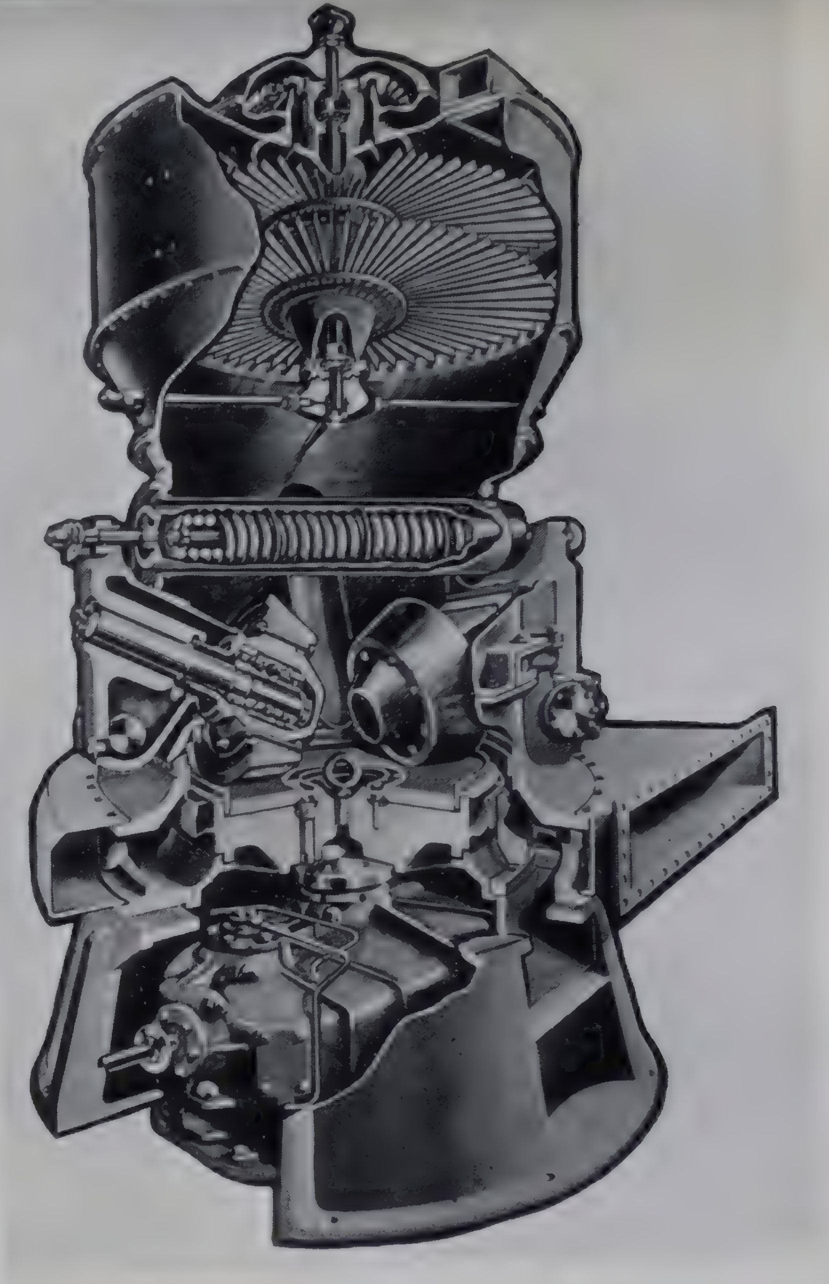


Fig. 24.29. Lopulco mill

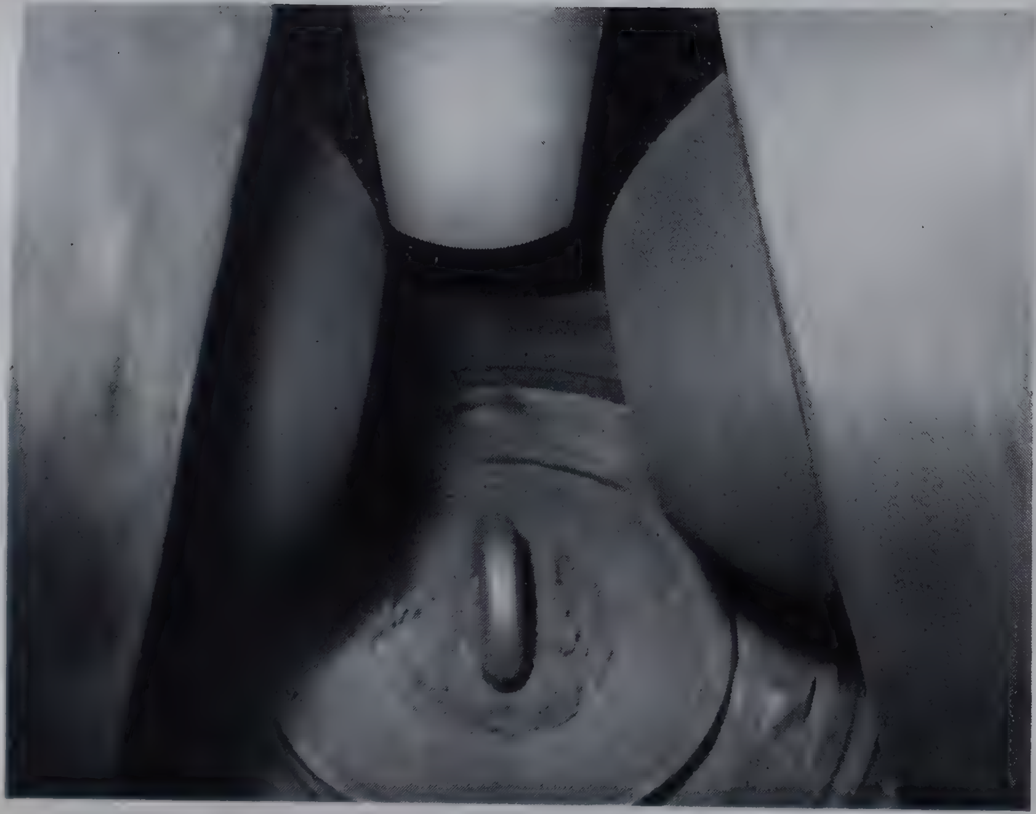


Fig. 24.30. Plan view of Lopulco mill showing rollers

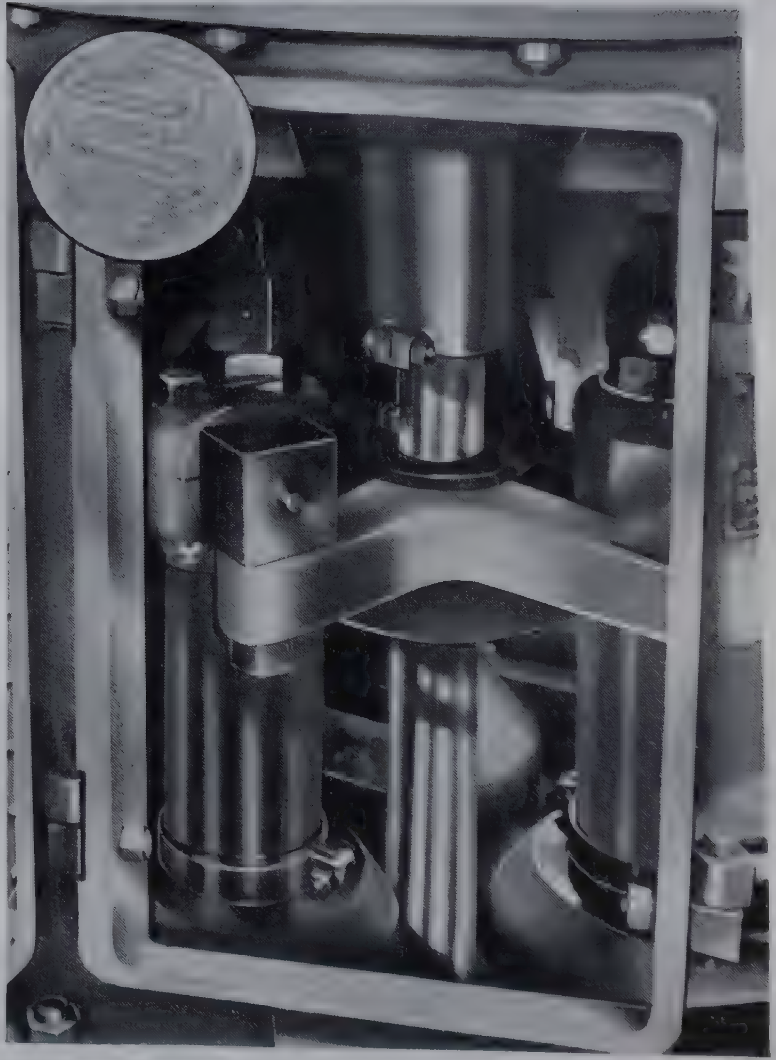
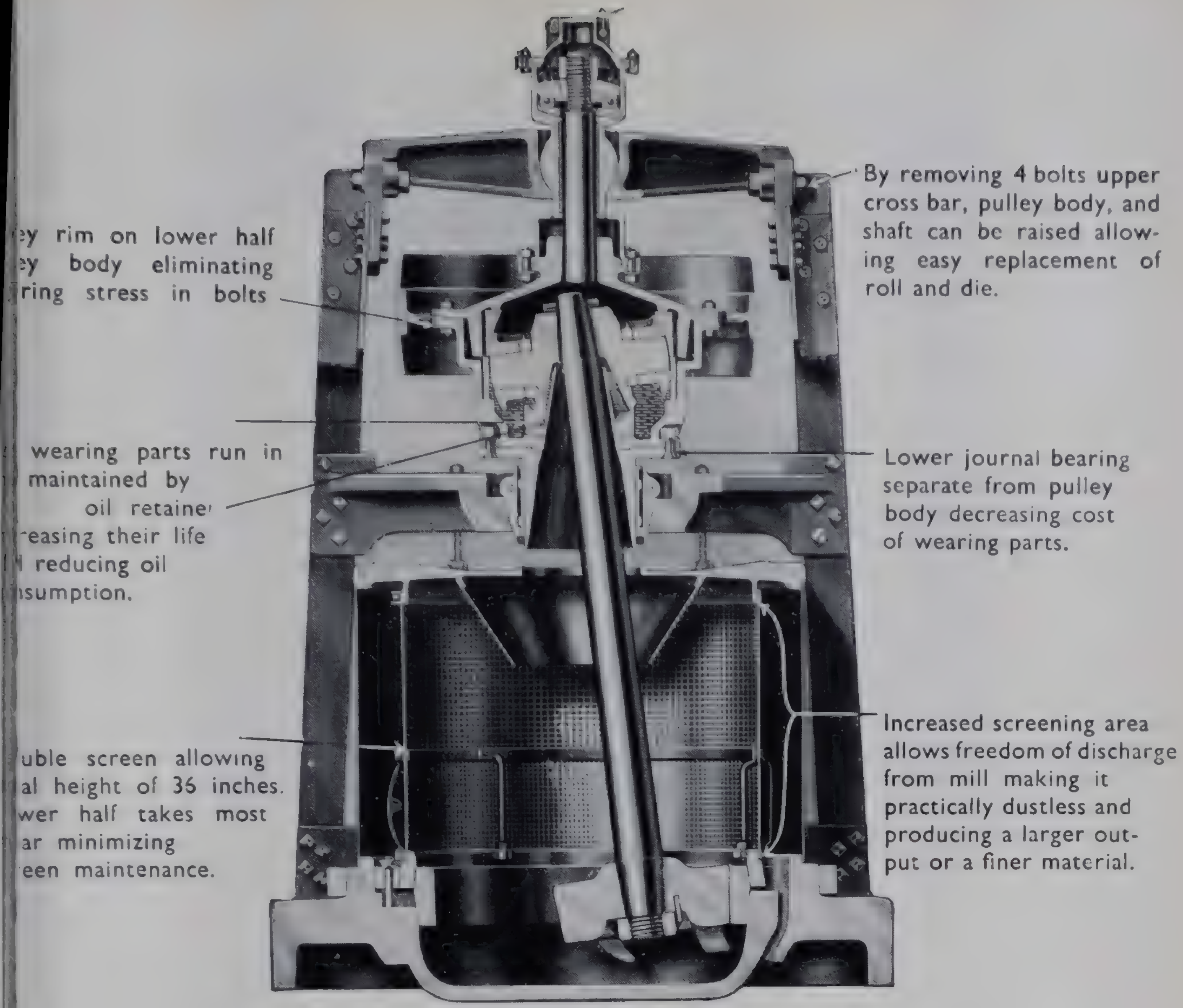


Fig. 24.31. Crushing heads of Raymond mill


ey rim on lower half
ey body eliminating
ring stress in bolts
wearing parts run in
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oil retaine
reasing their life
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uble screen allowing
al height of 36 inches.
wer half takes most
ar minimizing
een maintenance.
By removing 4 bolts upper cross bar, pulley body, and shaft can be raised allowing easy replacement of roll and die.
Lower journal bearing separate from pulley body decreasing cost of wearing parts.
Increased screening area allows freedom of discharge from mill making it practically dustless and producing a larger output or a finer material.

Fig. 24.32. Griffin mill

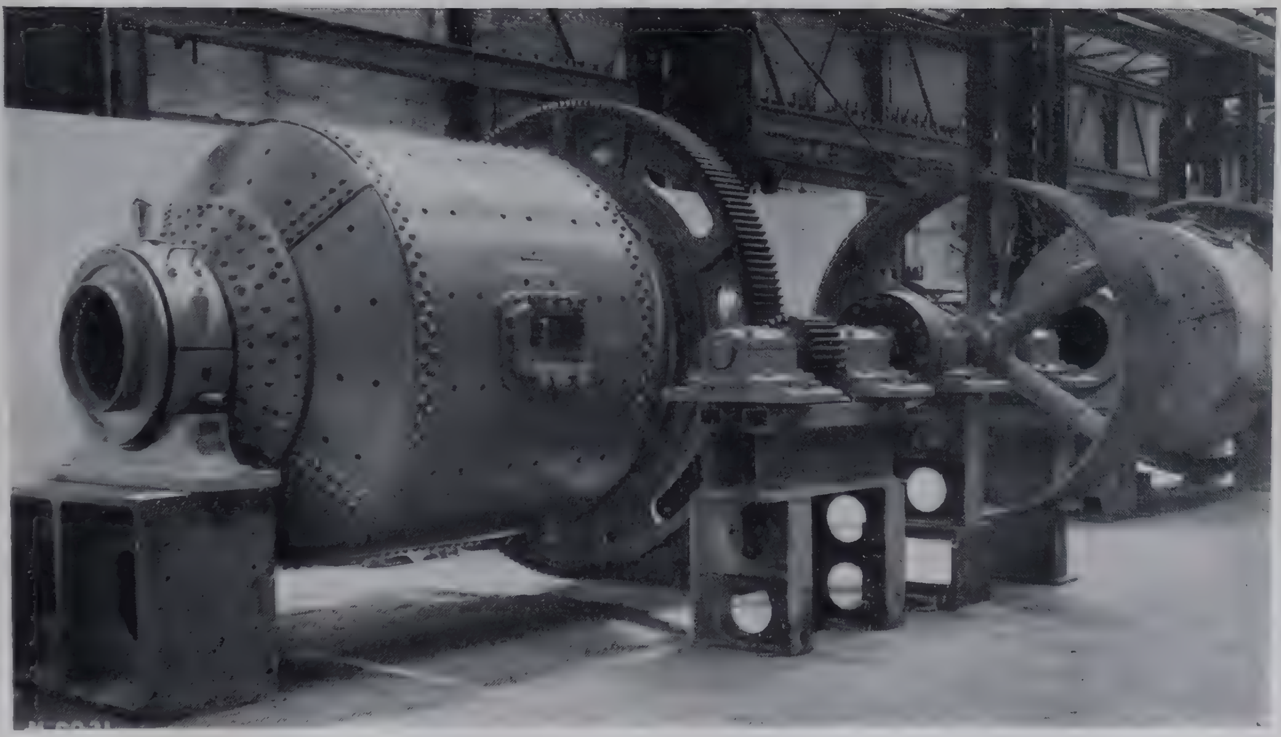

Fig. 24.33. A ball mill

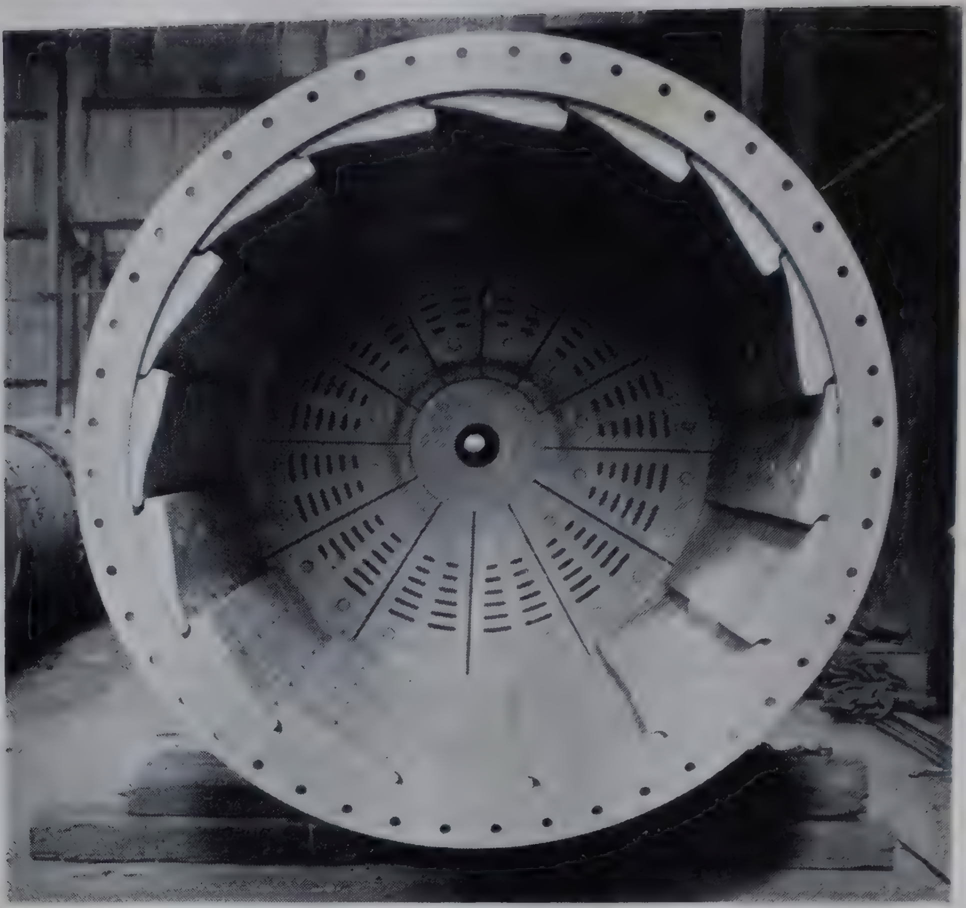

Fig. 24.34. View of inside of ball mill showing liners

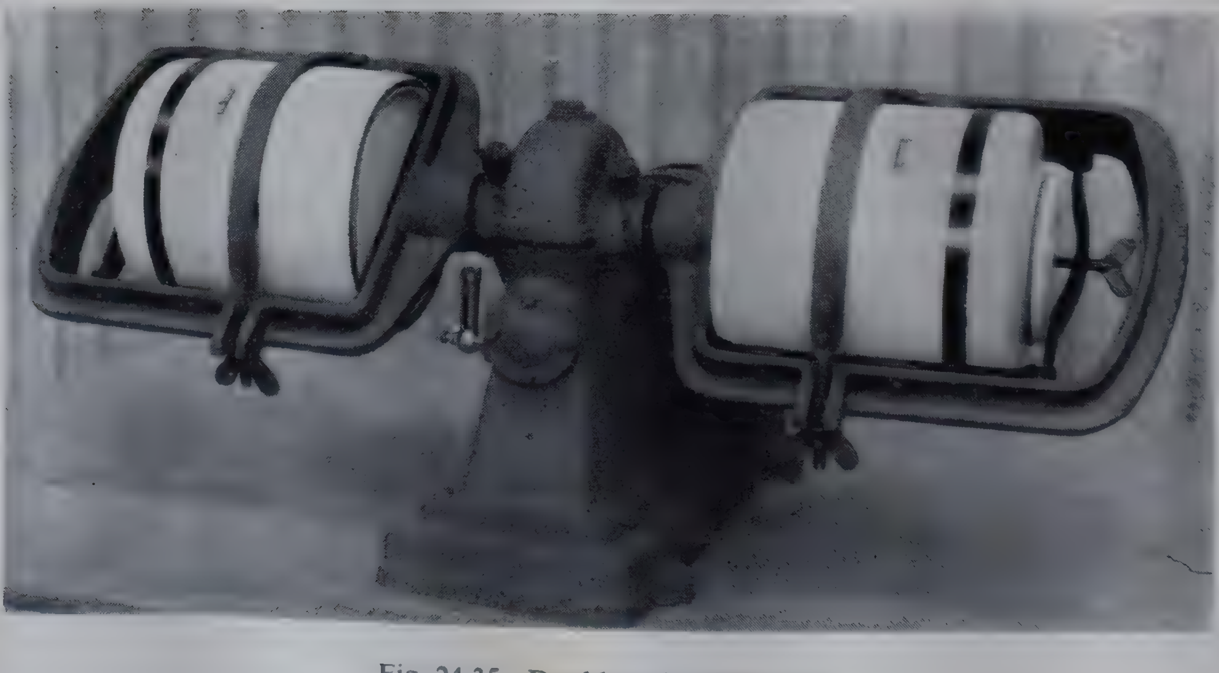

Fig. 24.35. Double unit pebble mill

***The Raymond Mill.*** The Raymond mill (Fig. 24.36) is slightly less economical in operation than the Lopulco mill, but will give a rather finer and more uniform product. A central shaft driven by a bevel gear carries a yoke at the top and terminates in a foot-step bearing at the bottom. On the yoke are pivoted a number of heavy arms (Fig. 24.31) carrying the grinding heads which are thrown outwards by centrifugal action and bear on a circular bull ring. Both the grinding heads and the bull ring are readily replaceable. The material, which is introduced by means of an automatic feed device, is forced on to the bull ring by means of a plough which rotates on the central shaft. The ground material is removed by means of an air current, as in the case of the Lopulco mill, and the oversize material falls back and is again brought on to the bull ring by the plough.

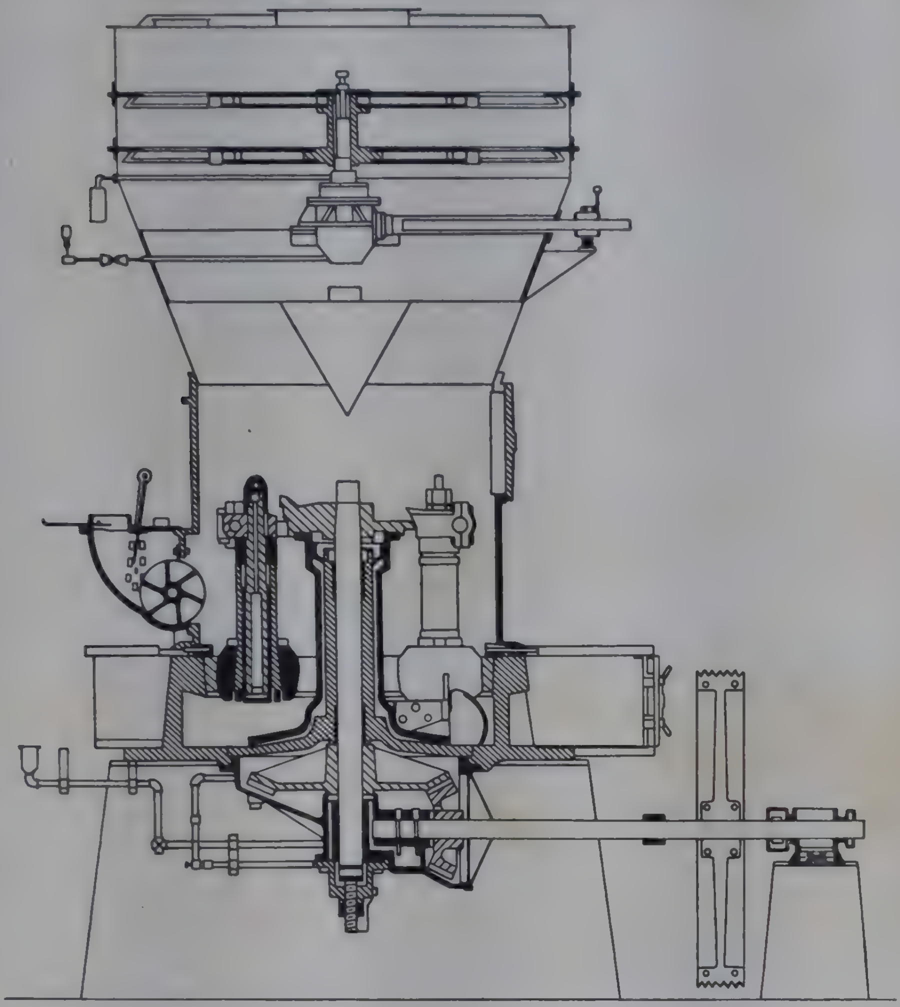

Fig. 24.36. Raymond mill

As the mill operates at high speeds, it is not suitable for use with abrasive materials; nor will it handle materials that soften during crushing. Though the power consumption and maintenance costs are low, this machine does not compare favourably with the Lopulco mill under most conditions. It has the added disadvantage that wear will take place if the machine is run without any feed, because no clearance is maintained between the grinding heads and the bull ring. Besides being used for the preparation of pulverised coal, the Raymond mill is used extensively in cement and pottery manufacture. In the pottery industry, sizing of the raw materials has to be carried out within very fine limits, possibly between 55 and 65 microns, and the Raymond mill is capable of achieving this. A comparison between the Lopulco and Raymond mills has shown that, whereas the Lopulco mill would give a product, 98% of which was below 300 mesh in size, the Raymond mill would give 100% below this size: in the latter case, however, the power consumption is considerably higher. Some typical figures for power consumption with the Raymond mill are given below.

| *Material* | *Feed Size* | *Size of Product* | *Power Consumption* H.P. Hours/ton |
|---|---|---|---|
| Coal | about $\frac{1}{8}''$ | 90% through 100 mesh | 19–20 |
| Limestone | dust-$\frac{1}{8}''$ | 99% through 100 mesh | |
| | | 90% through 200 mesh | 24 |
| Barytes | dust-$\frac{3}{4}''$ | 99% through 300 mesh | 52 |

### *The Crushing Force*

For a Raymond mill let

$M$ = the mass of the grinding head
$m$ = the mass per unit length of the arm
$\omega$ = the angular speed of rotation
$\theta$ = the angle between the arm and the vertical
$l$ = the length of the arm
$c$ = the radius of the yoke
$R$ = the normal reaction of the bull ring on the grinding head.

If we consider one arm rotating under uniform conditions, the sum of the moments of all the forces, acting on the roller and arm, about the point of support O will be zero (Fig. 24.37).

For a length d$y$ of the rod at a distance $y$ from O

mass of the element $= m\,\mathrm{d}y$

centrifugal force acting on element $= m\,\mathrm{d}y(c + y \sin\theta)\omega^2$

moment of the force about O $= -m\,\mathrm{d}y(c + y\sin\theta)\omega^2 y\cos\theta$

Total moment of the whole arm $= -m\omega^2\cos\theta(\frac{1}{2}cl^2 + \frac{1}{3}l^3\sin\theta)$

Moment of the centrifugal force acting on the grinding head $= -M\omega^2 l\cos\theta(c + l\sin\theta)$

Moment of the weight of the arm $= \frac{1}{2}ml^2 g\sin\theta$

Moment of the weight of the grinding head $= Mgl\sin\theta$

Moment of the normal reaction of the bull ring on the grinding head $= Rl\cos\theta$

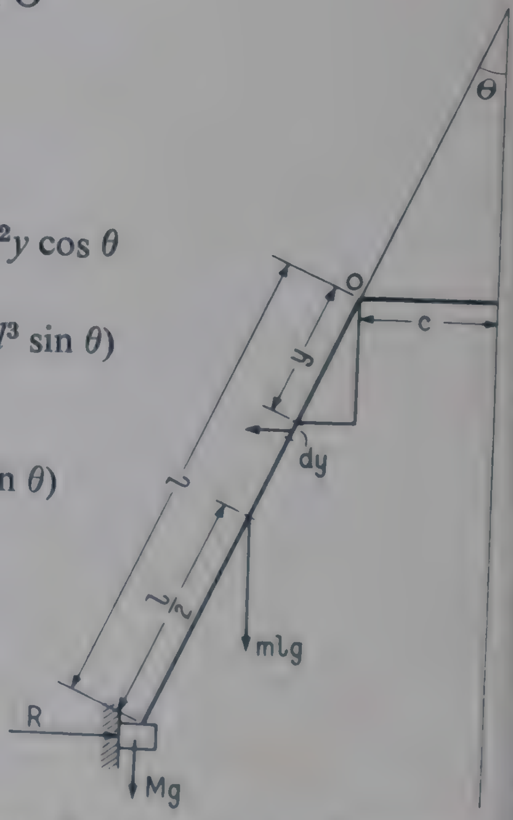

Fig. 24.37. Forces in Raymond mill

$$\text{Total moment about O} = \tfrac{1}{2}ml^2g\sin\theta + Mgl\sin\theta + Rl\cos\theta$$
$$- ml^2\omega^2\cos\theta(\tfrac{1}{2}c + \tfrac{1}{3}l\sin\theta) - M\omega^2l\cos\theta(c + l\sin\theta) = 0$$

Thus,

$$R = M'\omega^2(\tfrac{1}{2}c + \tfrac{1}{3}l\sin\theta) + M\omega^2(c + l\sin\theta) - \tfrac{1}{2}M'g\tan\theta - Mg\tan\theta$$

(where $M' = ml =$ mass of arm)

$$= M'(\tfrac{1}{2}\omega^2c + \tfrac{1}{3}l\omega^2\sin\theta - \tfrac{1}{2}g\tan\theta) + M(\omega^2c + \omega^2 l\sin\theta - g\tan\theta) \qquad \ldots.(24.14)$$

***The Griffin Mill.*** The Griffin mill (Fig. 24.32) is similar to the Raymond mill except that it employs only one grinding head and the separation of the product is effected using a screen.

## The Ball Mill

In its simplest form, the ball mill (Fig. 24.33) consists of a rotating hollow cylinder, partially filled with balls, with its axis either horizontal or at a small angle to the horizontal. The material to be ground may be fed in through a hollow trunnion at one end and the product leave through a similar trunnion at the other end. The outlet is normally covered with a coarse screen to prevent the escape of the balls.

The inner surface of the cylinder is usually lined with an abrasion resistant material (Fig. 24.34), such as manganese steel, stoneware or rubber. Less wear takes place in rubber lined mills, and the coefficient of friction between the balls and the cylinder is greater with steel or stoneware linings. The balls are therefore carried further in contact with the cylinder and thus drop on to the feed from a greater height. In some cases, lifter bars are fitted to the inside of the cylinder. A new type of ball mill is now being used to an increasing extent. The mill is vibrated instead of being rotated and the rate of passage of material is controlled by the slope of the mill.

The ball mill is used for the grinding of a wide range of materials, including coal, pigments, and felspar for pottery, and will take feed up to about 2 in. in size. The efficiency of grinding increases with the hold-up in the mill until the voids between the balls are filled. Further increase in the quantity then lowers the efficiency again.

***The Balls.*** The balls are usually made of flint or steel and occupy between 30 and 50% of the volume of the mill. The diameter of ball used will vary between ½″ and 5″ and the optimum diameter is approximately proportional to the square root of the size of the feed, the proportionality constant being a function of the nature of the material.

During grinding, the balls themselves wear and are constantly replaced by new ones so that the mill contains balls of various ages, and hence of various sizes. This is advantageous since the large balls deal effectively with the feed and the small ones are responsible for giving a fine product. The maximum rate of wear of steel balls, using very abrasive materials, is about 0.7 lb per ton of material for dry grinding, and 2 to 3 lb per ton for wet grinding. The normal charge of balls amounts to about 300 pounds per cubic foot. In small mills where very fine grinding is required, pebbles are often used in place of balls (Fig. 24.35).

In the compound mill, the cylinder is divided into a number of compartments by vertical perforated plates. The material flows axially along the mill and can pass from one compartment to the next only when its size has been reduced to less than that of the perforations in the plate. Each compartment is supplied with balls of a different size; the large balls are at the entry end and thus operate on the feed material, whilst the small balls come into contact with the material immediately before it is discharged. This results in economical operation and the formation of a uniform product.

***Factors Influencing the Size of the Product.*** 1. *The Rate of Feed.* With high rates of feed, less size reduction is effected since the material is in the mill for a shorter time.

2. *The Properties of the Feed Material.* The larger the feed the larger will be the product under given operating conditions. A smaller size reduction is obtained with a hard material.

3. *Weight of Balls.* A heavy charge of balls produces a fine product. The weight of the charge can be increased either by increasing the number of balls or by using a material of higher density. Since optimum grinding conditions are usually obtained when the bulk volume of the balls is equal to 50% of the volume of the mill, variation of the weight of balls is normally effected by the use of materials of different densities.

4. *The Diameter of the Balls.* Small balls facilitate the production of fine material but they do not deal so effectively with the larger particles in the feed. The limiting size reduction obtained with a given size of balls is known as the free grinding limit. For most economical operation, the smallest possible balls should be used.

5. *The Slope of the Mill.* Increase in the slope of the mill increases the capacity of the plant because the retention time is reduced but a coarser product is obtained.

6. *Discharge Freedom.* Increasing the freedom of discharge of the product has the same effect as increasing the slope. In some mills, the product is discharged through openings in the lining.

7. *The Speed of Rotation of the Mill.* At low speeds of rotation, the balls simply roll over one another and little crushing action is obtained. At slightly higher speeds, they are projected short distances across the mill, and at still higher speeds they are thrown greater distances and considerable wear of the lining of the mill takes place. At very high speeds, the balls are carried right

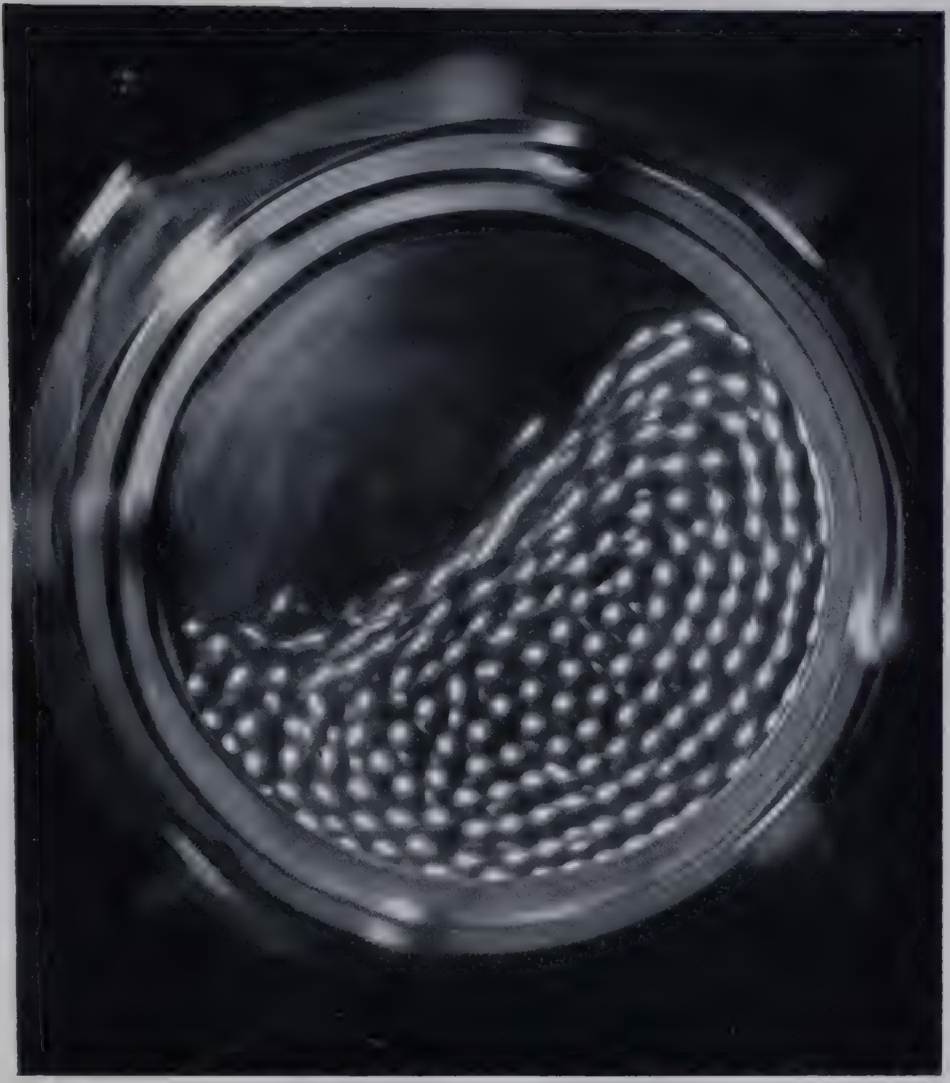

Fig. 24.38. A ball mill operating at correct speed

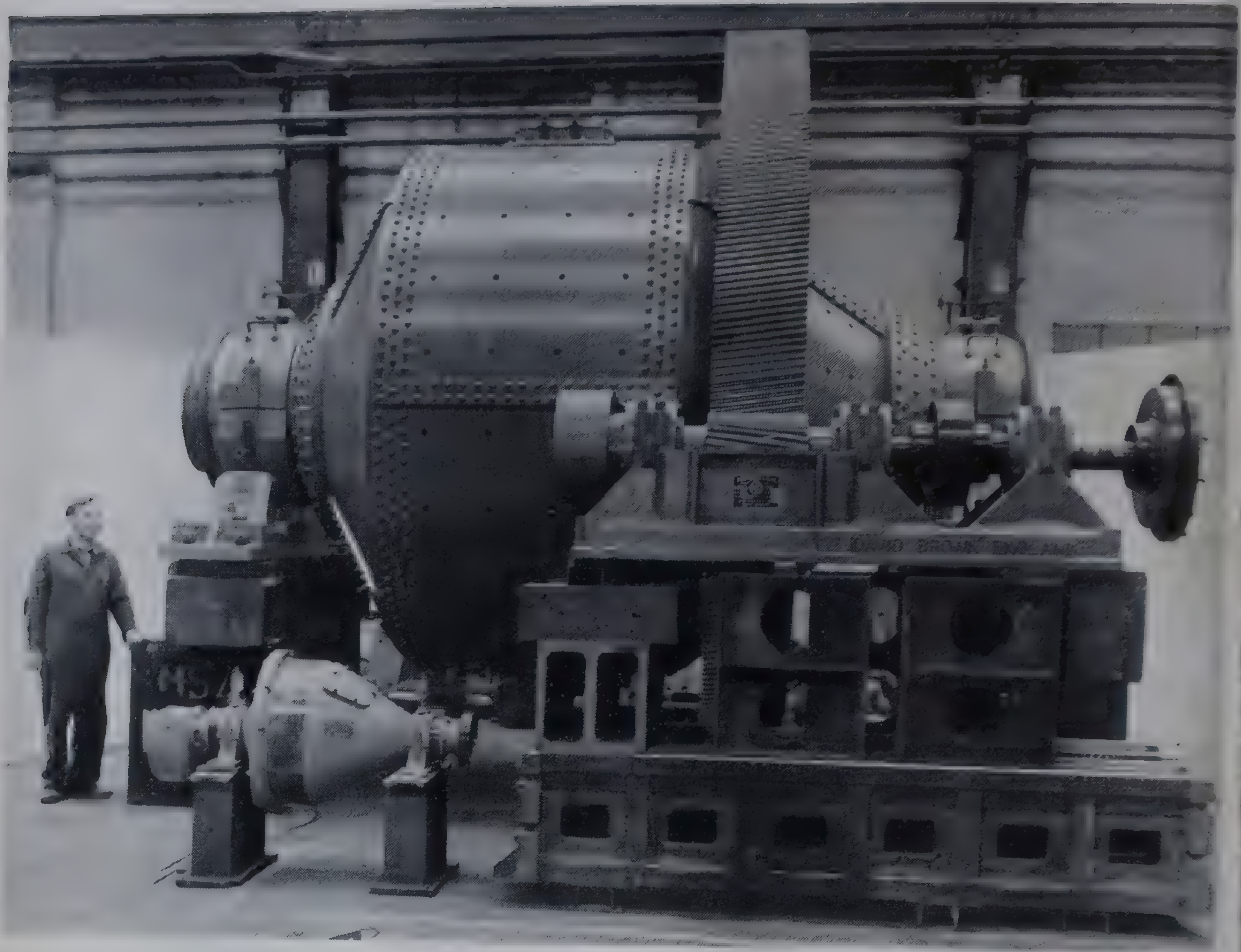

Fig. 24.40. Hardinge mill

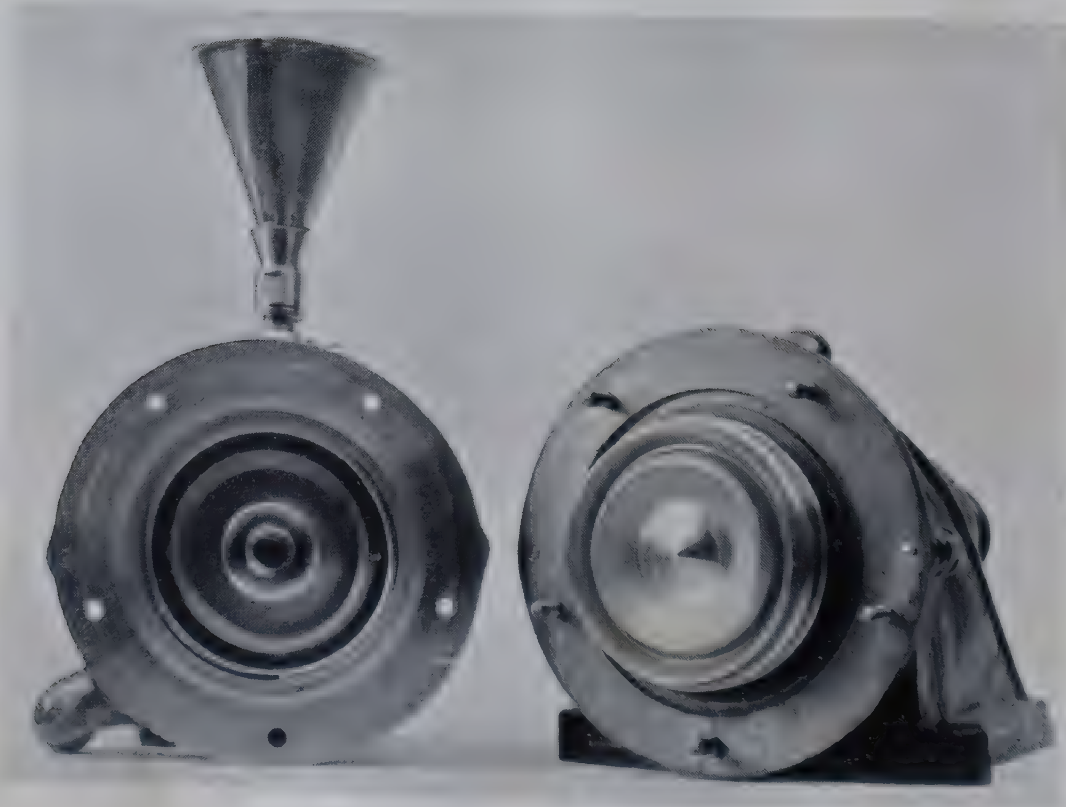

Fig. 24.41. Colloid mill

round in contact with the sides of the mill and little relative movement or grinding takes place again. The minimum speed at which the balls are carried round in this manner is called the critical speed of the mill and, under these conditions, there will be no resultant force acting on the ball when it is situated in contact with the lining of the mill in the uppermost position, i.e. the centrifugal force will be exactly equal to the weight of the ball. If the mill is rotating at the critical angular velocity $\omega_c$,

$$r \, . \, \omega_c^2 = g$$

i.e.

$$\omega_c = \sqrt{\frac{g}{r}} \qquad \ldots .(24.15)$$

Here $r$ is the radius of the mill less that of the particle. It is found that the optimum speed is between one half and three quarters of the critical speed. Fig. 24.38 illustrates conditions in a ball mill operating at the correct rate.

8. *The Level of Material in the Mill.* Power consumption is reduced by maintaining a low level of material in the mill, and this can be controlled most satisfactorily by fitting a suitable discharge opening for the product. If the level of material is raised, the cushioning action is increased and power is wasted by the production of an excessive quantity of undersize material.

***Advantages of the Ball Mill.*** 1. The mill can be used wet or dry but wet grinding facilitates the removal of the product.

2. The costs of installation and of power are low.

3. The ball mill can be used with an inert atmosphere and therefore can be used for the grinding of certain explosive materials.

4. The grinding medium is cheap.

5. The mill is suitable for materials of all degrees of hardness.

6. It can be used for batch or continuous operation.

7. It can be used for open or closed circuit grinding. With open circuit grinding a wide range of particle size is obtained in the product. With closed circuit grinding, the use of an external separator can be obviated by continuous removal of the product by means of a current of air or through a screen (Fig. 24.39).

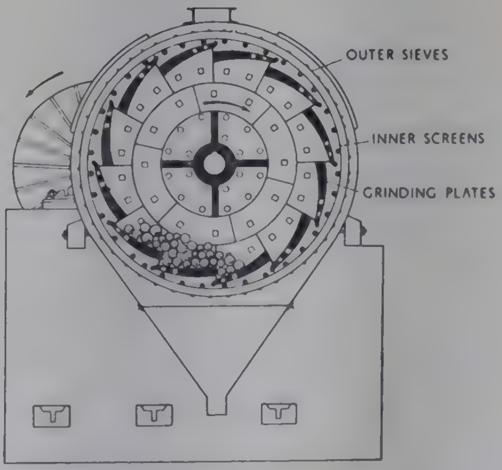

Fig. 24.39. End view of ball mill showing screens

## The Tube Mill

The tube mill is similar to the ball mill in construction and operation, but the ratio of the length to the diameter is usually 3 or 4 : 1, as compared with 1 or 1½ for the ball mill. The mill is filled with pebbles, rather smaller in size than the balls used in the ball mill, and the inside of the mill is so shaped th a layer of pebbles becomes trapped in it to form a self-renewing lining. Th characteristics of the two mills are similar but the material remains longer in th tube mill because of its greater length, and a finer product is therefore obtaine

### The Rod Mill

In the rod mill, high carbon steel rods about 2 in. diameter and extendin the whole length of the mill are used in place of balls. This mill gives a ver uniform fine product and power consumption is low, but it is not suitable fo very tough materials and the feed should not exceed about one inch in size Worn rods must be removed from time to time and replaced by new ones which are rather cheaper than balls. It is particularly useful with sticky material which would hold the balls together in aggregates because the greater weigh of the rods causes them to pull apart again.

### The Hardinge Mill

The Hardinge mill (Fig. 24.40) is a ball mill in which the balls segregat themselves according to size. The main portion of the mill is cylindrical, lik the ordinary ball mill, but the outlet end is conical and tapers towards the discharge point. The large balls collect in the cylindrical portion while the smaller balls, in order of decreasing size, locate themselves in the conical portion (Fig. 12.42). The material is therefore crushed by the action of successively smaller balls, and the mill is thus similar in characteristics to the compound ball mill. It is not known exactly how balls of different sizes segregate but it is suggested that, if the balls are initially mixed, the large ones will

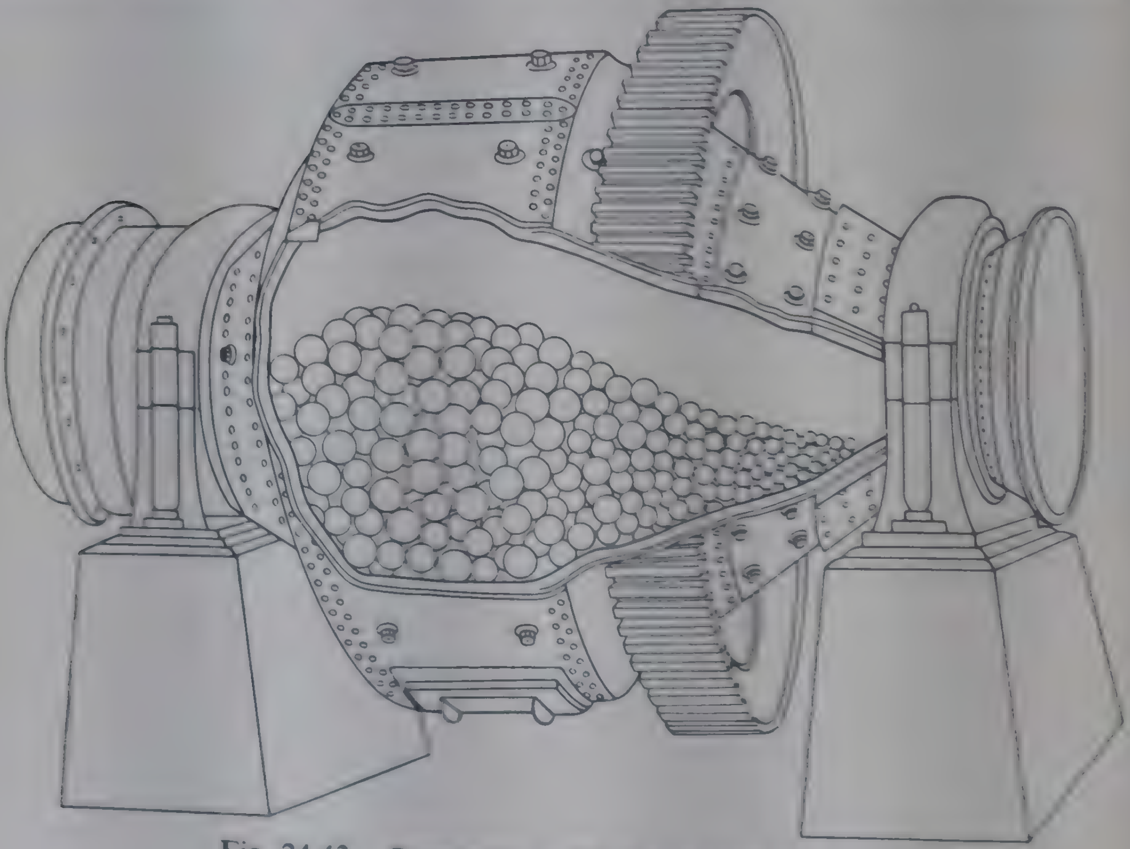

Fig. 24.42. Segregation of balls in Hardinge mill

ain a slightly higher falling velocity and therefore strike the sloping surface the mill before the smaller ones, and then run down towards the cylindrical ction. The mill has an advantage over the compound ball mill in that the rge balls are raised to the greatest height and therefore are able to exert the aximum force on the feed. As the size of the material is reduced, smaller rces are needed to cause fracture and it is therefore unnecessary to raise the naller balls so high. The capacity of the Hardinge mill is higher than that a ball mill of similar size and it gives a finer and more uniform product with lower consumption of power, but it is difficult to select an optimum speed ecause of the variation of diameter. It is extensively used for the grinding of ich materials as cement, fuels, carborundum, silica, talc, slate and barytes.

## COLLOID MILLS

olloidal suspensions and emulsions, with particle or droplet sizes less than one icron, are produced by means of a colloid mill. The mill shown in Fig. 24.41 onsists of a stator and a rotor with conical working surfaces between which nere is an adjustable clearance of between 2 and 30 thousandths of an inch. he rotor speed is between about 3000 and 15,000 r.p.m. The suspension, consisting of particles of about 100 mesh, is introduced from a funnel and is nrown outwards by centrifugal action, and size reduction is effected as it passes etween the working surfaces.

Another form of mill which does not give quite such a fine product is the jet ulveriser in which the solid is pulverised in jets of high pressure superheated team or compressed air, supplied at pressures up to 500 pounds per square inch. The pulverising takes place in a shallow cylindrical chamber with a number of ets arranged tangentially at equal intervals around the circumference. The olid is thrown to the outside walls of the chamber and the fine particles are ormed by the shearing action resulting from the differential velocities within he fluid streams. The jet pulveriser will give a product with a particle size between 1 and 10 microns.

In all colloid mills, the power consumption is very high and the material should therefore be ground as finely as possible before it is fed to the mill.

## CHAPTER 25

# *Classification of Solid Particles*

THE problem of separating solid particles according to their physical properties arises on a large scale in the mining industry, where it is necessary to separate the valuable constituents in a mineral from the adhering gangue, as it is called, which is usually of a lower density. In this case, it is first necessary to crush the material so that each individual particle contains only one constituent. The coal mining industry also is faced with a similar problem in coal washing plants in which dirt is separated from the clean coal. The chemical industry is more usually concerned with separating a single material, for instance, the product from a size reduction plant, into a number of size fractions, or to obtain a uniform material for incorporation in a system in which a chemical reaction takes place. As similar problems are involved in separating a mixture into its constituents and into size factors, the two processes will here be considered together.

Separation depends on the selection of a process in which the behaviour of the material is influenced to a very marked degree by some physical property. Thus, if a material is to be separated into various size fractions, a sieving method may be used because this process depends primarily on the size of the particles, though other physical properties such as the shape of the particles and their tendency to agglomerate may also be involved. Other methods of separation depend on the differences in the behaviour of the particles in a moving fluid, and in this case the size and the density of the particles are the most important factors and shape is of secondary importance. Other processes make use of differences in electrical or magnetic properties of the materials or in their surface properties.

Generally, large particles are separated into size fractions by means of screens and small particles, which would clog the fine apertures of the screen or for which it would be impracticable to make the openings sufficiently fine, are separated in a fluid. Fluid separation is commonly used for separating a mixture of two materials but magnetic, electrostatic and froth flotation methods are also used where appropriate.

### Separation of Particles in a Fluid

Most processes which depend on differences in the behaviour of particles in a stream of fluid separate materials according to their terminal falling velocities, which in turn depend primarily on density and size and to a lesser extent on shape. Thus, in many cases it is possible to use the method to separate a mixture of two materials into its constituents, or to separate a mixture of particles of the same material into a number of size fractions.

Suppose that it is desired to separate particles of a relatively dense material

$A$ from particles of a less dense material $B$. If the size range is large, the terminal falling velocities of the largest particles of $B$ may be greater than those of the smallest particles of $A$, and therefore a complete separation will not be possible. The maximum range of sizes that can be separated is calculated from the ratio of the sizes of the particles of the two materials which have the same terminal falling velocities. If streamline conditions exist in the fluid, the terminal falling velocity of a particle of $A$ of equivalent spherical diameter $d_A$ is given from equation 14.16 as

$$u_{OA} = \frac{d_A^2 g}{18\mu}(\rho_A - \rho) \qquad \ldots.(25.1a)$$

and the terminal falling velocity of a particle of $B$ of size $d_B$ is given by

$$u_{OB} = \frac{d_B^2 g}{18\mu}(\rho_B - \rho) \qquad \ldots.(25.1b)$$

where $u_{OA}$ is the terminal falling velocity of the particle of $A$,
$u_{OB}$ is the terminal falling velocity of the particle of $B$,
$\rho_A$ is the density of material $A$,
$\rho_B$ is the density of material $B$,
$\rho$ is the density of the fluid,
$\mu$ is the viscosity of the fluid, and
$g$ is the acceleration due to gravity.

If the terminal falling velocities of the two particles are the same,

$$\frac{d_B}{d_A} = \left\{\frac{\rho_A - \rho}{\rho_B - \rho}\right\}^{\frac{1}{2}} \qquad \ldots.(25.2)$$

If the conditions in the fluid are turbulent, the terminal velocities are

$$u_{OA} = \sqrt{\frac{3d_A g}{\rho}(\rho_A - \rho)} \qquad \ldots.(25.3a)$$

and

$$u_{OB} = \sqrt{\frac{3d_B g}{\rho}(\rho_B - \rho)} \qquad \ldots.(25.3b)$$

(from equation 14.17)

For equal terminal falling velocities under these conditions,

$$\frac{d_B}{d_A} = \frac{\rho_A - \rho}{\rho_B - \rho} \qquad \ldots.(25.4)$$

Thus, separation is possible only if the ratio of the size of the largest particle of $B$ to that of the smallest particle of $A$ is less than

$$\frac{d_B}{d_A} = \left\{\frac{\rho_A - \rho}{\rho_B - \rho}\right\}^{s} \qquad \ldots.(25.5)$$

where $s = \frac{1}{2}$ for settling under streamline conditions and
$s = 1$ for settling under turbulent conditions.

For conditions that are not fully turbulent, $s$ lies between $\frac{1}{2}$ and 1.

It is seen that this size range becomes wider with increase in the density of the separating fluid and, when the fluid has the same density as the less dense material, complete separation is possible whatever the relative sizes. Although water is the most commonly used fluid, a specific gravity greater than unity is obtained if hindered settling takes place. Frequently, the fluid density is increased artificially by forming a suspension of small particles of a very dense solid, such as galena, ferro-silicon, magnetite, sand or clay, in the water. These suspensions have effective specific gravities up to about 3.5. Alternatively zinc chloride or calcium chloride solutions may be used. On the small scale liquids with a range of densities are produced by mixing benzene and carbon tetrachloride.

**Example.** It is desired to separate into two pure fractions a mixture of quartz and galena of a size range from 0·00058 to 0·00250 in. by the use of a hindered settling process. What is the minimum apparent density of the fluid that will give this separation? How will the viscosity of the bed affect the minimum required density? (Density of galena = 7·5 gm/cc. Density of quartz = 2·65 gm/cc.)

**Solution.** Assume shapes of galena and quartz particles are similar. From equation 25.2, required density of fluid for viscous conditions is given by

$$\frac{0{\cdot}00250}{0{\cdot}00058} = \left\{\frac{7{\cdot}5 - \rho}{2{\cdot}65 - \rho}\right\}^{\frac{1}{2}}$$

$$\therefore \qquad \rho = 2{\cdot}37 \text{ gm/cc}$$

From equation 25.4, required density of fluid for fully turbulent conditions is given by

$$\frac{0{\cdot}00250}{0{\cdot}00058} = \frac{7{\cdot}5 - \rho}{2{\cdot}65 - \rho}$$

$$\therefore \qquad \rho = 1{\cdot}18 \text{ gm/cc}$$

Thus the required density of the fluid is between $\underline{\underline{1{\cdot}18}}$ and $\underline{\underline{2{\cdot}37}}$ according to the value of the Reynolds Group, and hence according to the viscosity.

If the particles are allowed to settle in the fluid for only a very short time, they will not attain their terminal falling velocities and a better degree of separation can be obtained. A particle of material $A$ will have an initial acceleration $g\left(1 - \frac{\rho}{\rho_A}\right)$, because there is no fluid friction when the relative velocity is zero. Thus the initial velocity is a function only of density and is unaffected by size and shape. A very small particle of the denser material will therefore always commence settling at a greater rate than a large particle of the less dense material. Theoretically, therefore, it should be possible to separate materials completely, irrespective of the size range, provided that the periods of settling are sufficiently short. In practice, the required periods will often be so short that it is impossible to make use of this principle alone, but a better degree of separation can be obtained if the particles are not allowed to become fully accelerated. As the time of settling increases the larger particles of the less dense material catch up and overtake the small heavy particles.

Size separation equipment in which particles move in a fluid stream will now be described. Most of the plant utilises the difference in the terminal falling velocities of the particles; in the hydraulic jig, however, the particles are allowed to settle for only very brief periods at a time, and this equipment can therefore be used when the size range of the material is large.

***The Settling Tank.*** The material is introduced in suspension into a tank containing a relatively large volume of water moving at a low velocity as shown in Fig. 25.1. The particles soon enter the slowly moving water and, because the small particles settle at a lower rate, they are carried further forward before they reach the bottom of the tank; the very fine particles are carried away in the liquid overflow. Receptacles at various distances from the inlet collect different grades of particles according to their terminal falling velocities, the particles of high terminal falling velocity collecting near the inlet. The positions at which the particles are collected can be calculated on the assumption that they rapidly reach their terminal falling velocities, and attain the same horizontal velocity as the fluid.

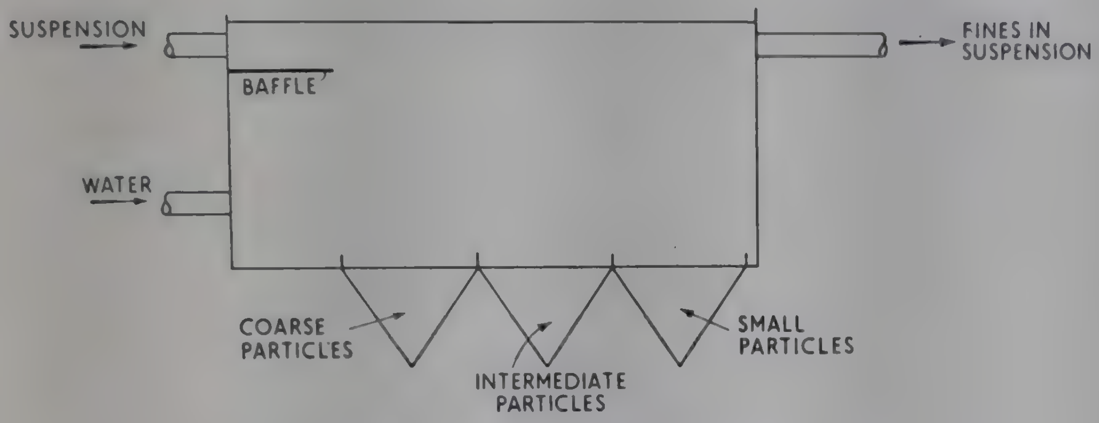

Fig. 25.1. Gravity settling tank

If the material is introduced in solid form down a chute, the position at which the particles are deposited will be determined by the rate at which they lose the horizontal component of their velocities. The larger particles will therefore be carried further forward and the smaller particles will be deposited near the inlet. The position at which the particles are deposited can be calculated using the relations given in Chapter 14.

***The Elutriator.*** Small quantities of material can be separated in the laboratory by means of an elutriator, which consists of a vertical tube up which fluid is passed at a controlled velocity. The particles are introduced, often through a side tube, and the smaller particles are carried over in the fluid stream while the large particles settle against the upward current. Further size fractions can be collected if the overflow from the first tube is passed vertically upwards through a second tube of greater cross-section; any number of such tubes can be arranged in series. The concentrations which are used in the elutriator are so low that settling takes place under approximately free settling conditions, and the method is therefore capable of adaptation for the determination of particle sizes. Details of the method for carrying out size analyses are given in British Standard 893[22], and Fig. 25.2 shows the standard elutriator for particles with settling velocities between 0.7 and 7.0 cm/sec.

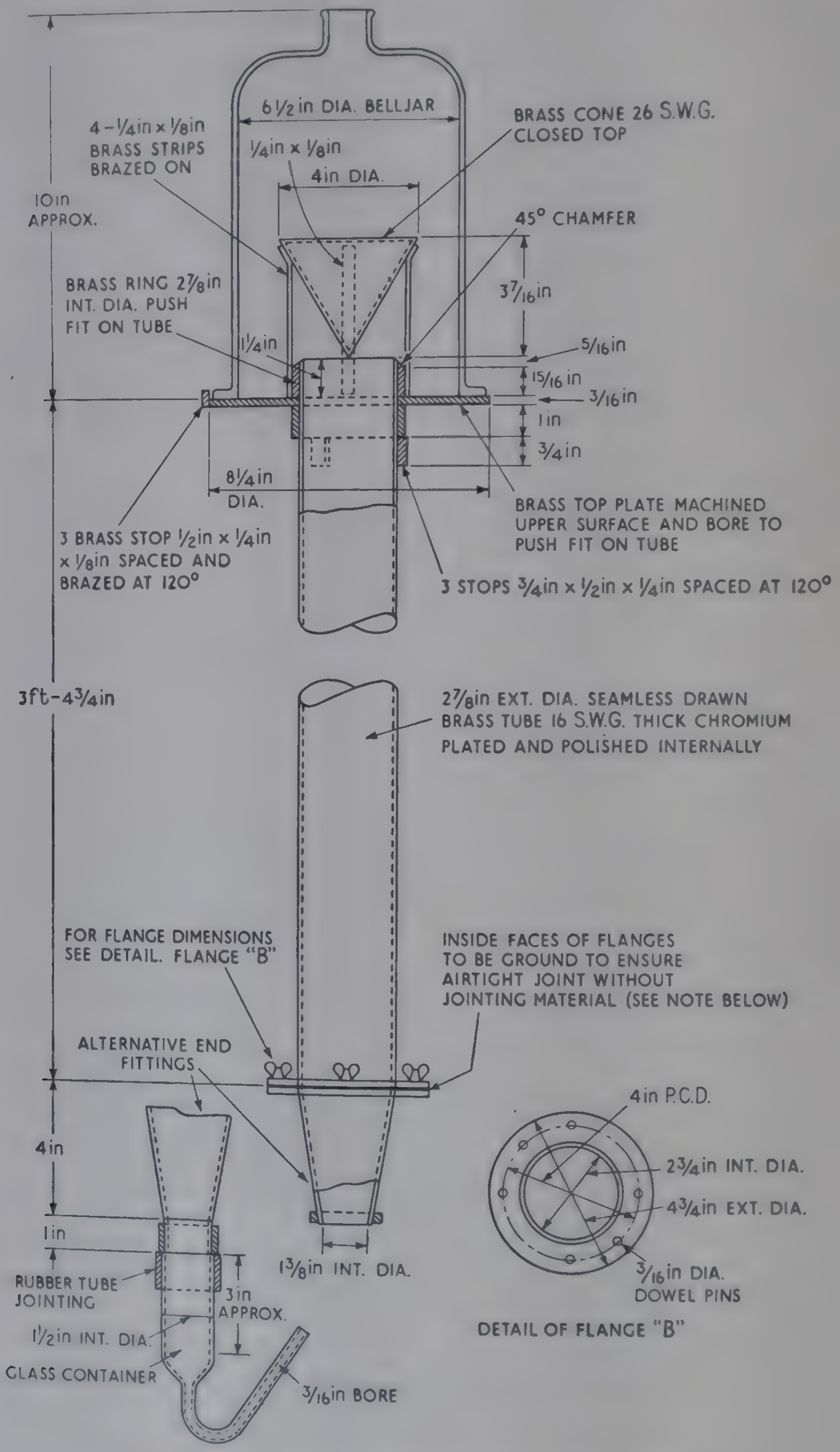
6½in DIA. BELLJAR
4-¼in × ⅛in BRASS STRIPS BRAZED ON
¼in × ⅛in
4in DIA.
BRASS CONE 26 S.W.G. CLOSED TOP
10in APPROX.
45° CHAMFER
3 7/16in
BRASS RING 2⅞in INT. DIA. PUSH FIT ON TUBE
1¼in
5/16in
15/16 in
3/16in
1in
¾in
8¼in DIA.
BRASS TOP PLATE MACHINED UPPER SURFACE AND BORE TO PUSH FIT ON TUBE
3 BRASS STOP ½in × ¼in × ⅛in SPACED AND BRAZED AT 120°
3 STOPS ¾in × ½in × ¼in SPACED AT 120°
3ft-4¾in
2⅞in EXT. DIA. SEAMLESS DRAWN BRASS TUBE 16 S.W.G. THICK CHROMIUM PLATED AND POLISHED INTERNALLY
FOR FLANGE DIMENSIONS SEE DETAIL. FLANGE "B"
INSIDE FACES OF FLANGES TO BE GROUND TO ENSURE AIRTIGHT JOINT WITHOUT JOINTING MATERIAL (SEE NOTE BELOW)
ALTERNATIVE END FITTINGS
4in
1in
RUBBER TUBE JOINTING
3in APPROX.
1⅜in INT. DIA.
1½in INT. DIA.
GLASS CONTAINER
3/16in BORE
4in P.C.D.
2¾in INT. DIA.
4¾in EXT. DIA.
3/16in DIA. DOWEL PINS
DETAIL OF FLANGE "B"

Fig. 25.2. Standard elutriator with 2¾ in. tube

***The Spitzkasten.*** The plant consists of a series of vessels of conical shape arranged in series. A suspension of the material is fed into the top of the first vessel and the larger particles settle, while the smaller ones are carried over in the liquid overflow and enter the top of a second conical vessel of greater cross-sectional area. The bottoms of the vessels are fitted with wide diameter outlets, and a stream of water can be introduced near the outlet so that the particles have to settle against a slowly rising stream of liquid. The size of the smallest particle which is collected in each of the vessels is influenced by the upward velocity at the bottom outlet of each of the vessels. The size of each successive vessel is increased, partly because the amount of liquid to be handled includes all the water used for classifying in the previous vessels, and partly because it is desired to reduce, in stages, the surface velocity of the fluid flowing from one vessel to the next. The Spitzkasten thus combines the principles used in the settling tank and in the elutriator.

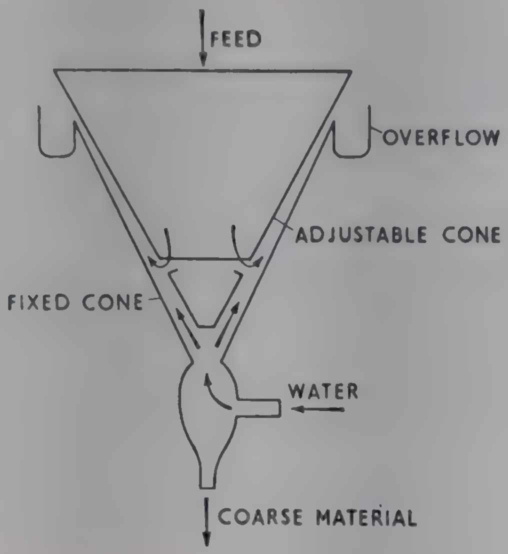

Fig. 25.3. Double cone classifier

The size of the material collected in each of the units is determined by the rate of feeding of suspension, the upward velocity of the liquid in the vessel and the diameter of the vessel. The equipment can also be used for separating a mixture of materials into its constituents, provided that the size range is not large. The individual units can be made of wood or sheet metal.

The Dorrco Sizer, illustrated in Fig. 25.5, works on the same principle but has a number of compartments trapezoidal in section. It is suitable for use with materials finer than about 4-mesh and it works at high concentrations in order to obtain the advantages of hindered settling.

***The Double Cone Classifier.*** This classifier (Fig. 25.3) consists of a conical vessel, with a second hollow cone of greater angle arranged apex downwards inside it so that there is an annular space of approximately constant cross section between the two cones. The bottom portion of the inner cone is cut away and its position relative to the outer cone can be regulated by a screw adjustment. Water is passed in an upward direction, as in the Spitzkasten, and overflows into a launder arranged round the whole of the periphery of the outer cone. The material to be separated is fed in suspension to the centre of the inner cone, and the liquid level is maintained slightly higher than the overflow level, so that there is a continuous flow of liquid downwards in the centre cone. The particles are therefore brought into the annular space where they are subjected to a classifying action; the smaller particles are carried away in the overflow, and the larger particles settle against the liquid stream and are taken off at the bottom.

***The Rake Classifier.*** Several forms of classifier exist in which the material of lower settling velocity is carried away in a liquid overflow, and the material of higher settling velocity is deposited on the bottom of the equipment and is dragged upwards against the flow of the liquid, by some mechanical means. During the course of the raking action, the solids are turned over so that any small particles trapped under larger ones are brought to the top again.

The Dorr classifier, which consists of a shallow rectangular tank inclined to the horizontal, has already been referred to in Chapter 17 and is illustrated in Fig. 17.5. The feed is introduced in the form of a suspension near the middle of the tank and water for classifying is added at the upper end. The liquid, together with the material of low terminal falling velocity, flows down the tank under a baffle and is then discharged over an overflow weir. The heavy material settles to the bottom and is then dragged upwards by means of a rake; it is thus separated from the liquid and is discharged at the upper end of the tank. The Dorr classifier is extensively used in conjunction with ball mills where it is often necessary to separate a large amount of fines from the oversize material. Arrangement of a number of Dorr classifiers in series enables a material to be separated into several size fractions.

Numerous other classifiers of a similar type are made; they include the Stokes double acting classifier (Fig. 25.6), in which a more uniform discharge of the heavy solids is obtained by the use of two sets of rakes suitably staggered.

The Akins classifier and the Denver cross-flow classifier (Fig. 25.7) are similar in action but employ a trough which is semi-circular in cross section, and the material which settles to the bottom is continuously moved to the upper end by means of a rotating helical scraper.

***The Bowl Classifier.*** The bowl classifier, which is used for fine materials, consists of a shallow bowl with a concave bottom (Fig. 25.8). The suspension is fed into the centre of the bowl near the liquid surface, and the liquid and the fine particles are carried in a radial direction and pass into the overflow, an open launder running round the whole of the periphery of the bowl at the top. The heavier or larger material settles to the bottom and is raked towards the outlet at the centre. The classifier has a large overflow area, and consequently high volumetric rates of flow of liquid can be used without producing a high linear velocity at the overflow. Its action will be seen to be similar to that of a thickener which is effecting incomplete clarification.

***The Hydraulic Jig.*** The hydraulic jig operates by allowing material to settle for brief periods so that the particles do not attain their terminal falling velocities, and is therefore suitable for separating materials of wide size range into their constituents. The material to be separated is fed dry, or more usually in suspension, over a screen and is subjected to a pulsating action by liquid which is set in oscillation by means of a reciprocating plunger. The particles on the screen constitute a suspension of high concentration and therefore the advantages of hindered settling are obtained. The jig usually consists of a rectangular-section tank with a tapered bottom, divided into two portions by a vertical baffle. In one section, the plunger operates in a vertical direction; the other incorporates the screen over which the separation is carried out. In addition, a stream of liquid is fed to the jig during the upward stroke.

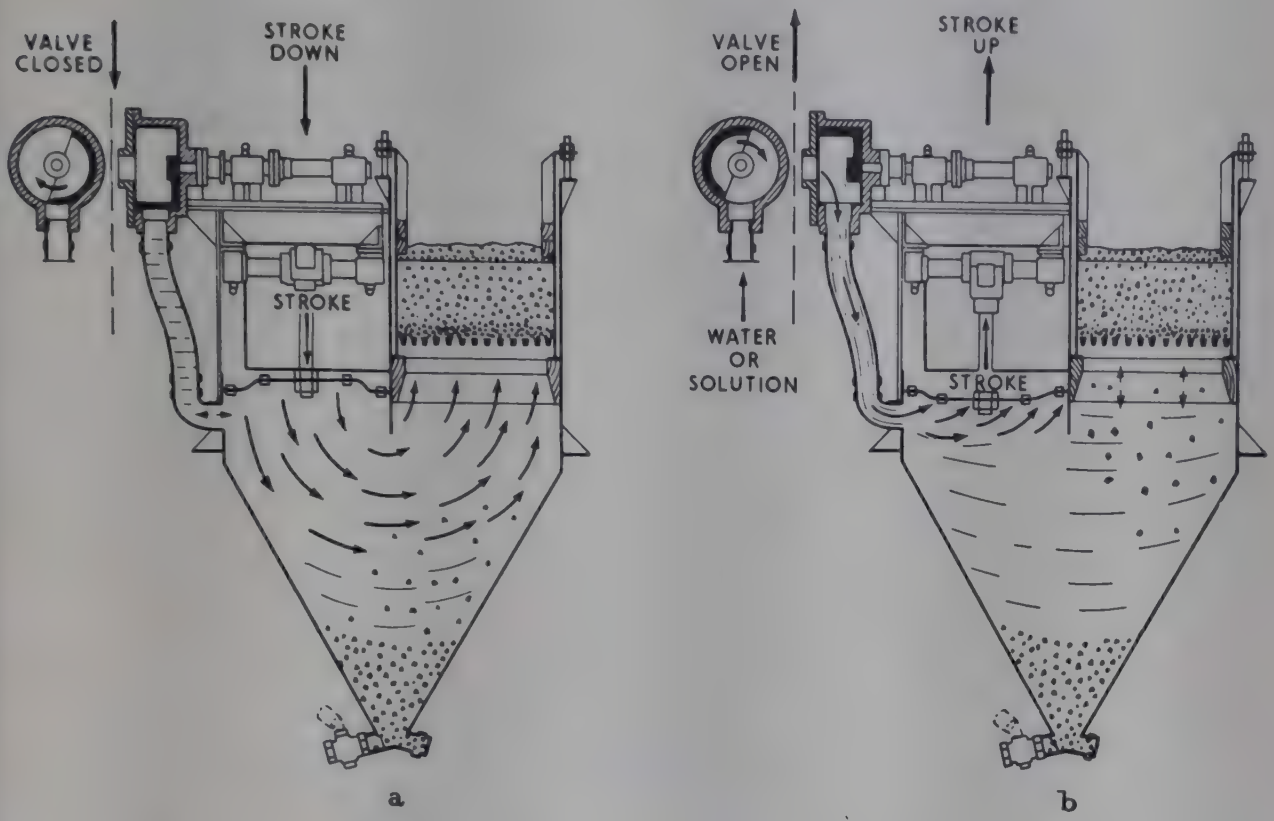

Fig. 25.4. Hydraulic jig. (*a*) Downward stroke (*b*) Upward stroke

The particles on the screen are brought into suspension during the downward stroke of the plunger (Fig. 25.4*a*) and are then allowed to settle during the upward stroke when the input of water is adjusted so that there is virtually no flow through the bed (Fig. 25.4*b*). The period of the plunger is small so that the denser material tends to collect near the screen and the lighter material above it. After a short time the material becomes divided into three strata, the bottom layer consisting of the large particles of the heavy material, the next of large particles of the lighter material together with small particles of the heavy material, and the top stratum of small particles of the light material. Large particles wedge at an earlier stage than small ones and therefore the small particles of the denser material are able to fall through the spaces between the larger particles of the light material. Many of these small particles then fall through the supporting gauze.

Four separate fractions are obtained from the jig and the successful operation of the plant depends on their rapid removal. The small particles of the heavy material which have fallen through the gauze are taken off at the bottom of the tank. The larger particles of each of the materials are retained on the gauze in two layers; the denser material at the bottom and the less dense material on top. These two fractions are removed through gates at the side of the jig. The remaining material, consisting of small particles of the light material, is carried away in the liquid overflow.

### Riffled Tables

The riffled table, of which the Wilfley table (Fig. 25.9) is a typical example, consists of a flat table which is inclined at an angle of about 3° to the horizontal. Running approximately parallel to the top edge are a series of slats, or riffles as they are termed, about $\frac{1}{4}$ in. in height. The material to be separated is fed on to one of the top corners and a reciprocating motion, consisting of a slow movement in the forward direction and a very rapid return stroke causes it to move across the table. The particles also tend to move downwards under the combined action of gravity and of a stream of water which is introduced along the top edge, but are opposed by the riffles behind which the smaller particles or the denser material tend to be held. Thus, the large particles and the less dense material are carried downwards, and the remainder is carried parallel to the riffles. In many cases, each riffle is tapered with the result that a number of fractions can be collected. Riffled tables can be used for separating materials down to about 300 mesh in size, provided that the difference in the densities is large.

### Centrifugal Separators

The use of cyclone separators for the removal of suspended dust particles from gases has already been referred to in Chapter 16, and a method has been given for calculating the size of the smallest particle which will be retained in the separator. By suitable choice of operating conditions, it is possible to use centrifugal methods for the classification of solid particles according to their terminal falling velocities. In general, the equipment is a little more elaborate than the simple cyclone separator. Fig. 25.11 shows a typical air separation unit which is similar in construction to the double cone classifier. The solids are fed to the bottom of the annular space between the cones and are carried upwards in the air stream and enter the inner cone through a series of ports fitted with adjustable vanes. As will be seen in the diagram, the suspension enters approximately tangentially and is therefore subjected to the action of centrifugal force. The coarse solids are thrown outwards against the walls and fall to the bottom under the action of gravity, while the small particles are removed by means of an exhaust fan. This type of separator is widely used for separating the oversize material from the product from a ball mill, and is suitable for materials as fine as 300 mesh.

A mechanical air separator is shown in Fig. 25.10. The material is introduced at the top through the hollow shaft and falls on to the rotating disc which throws it outwards. Very large particles fall into the inner cone and the remainder are lifted by the air current produced by the rotating vanes above the disc. Because

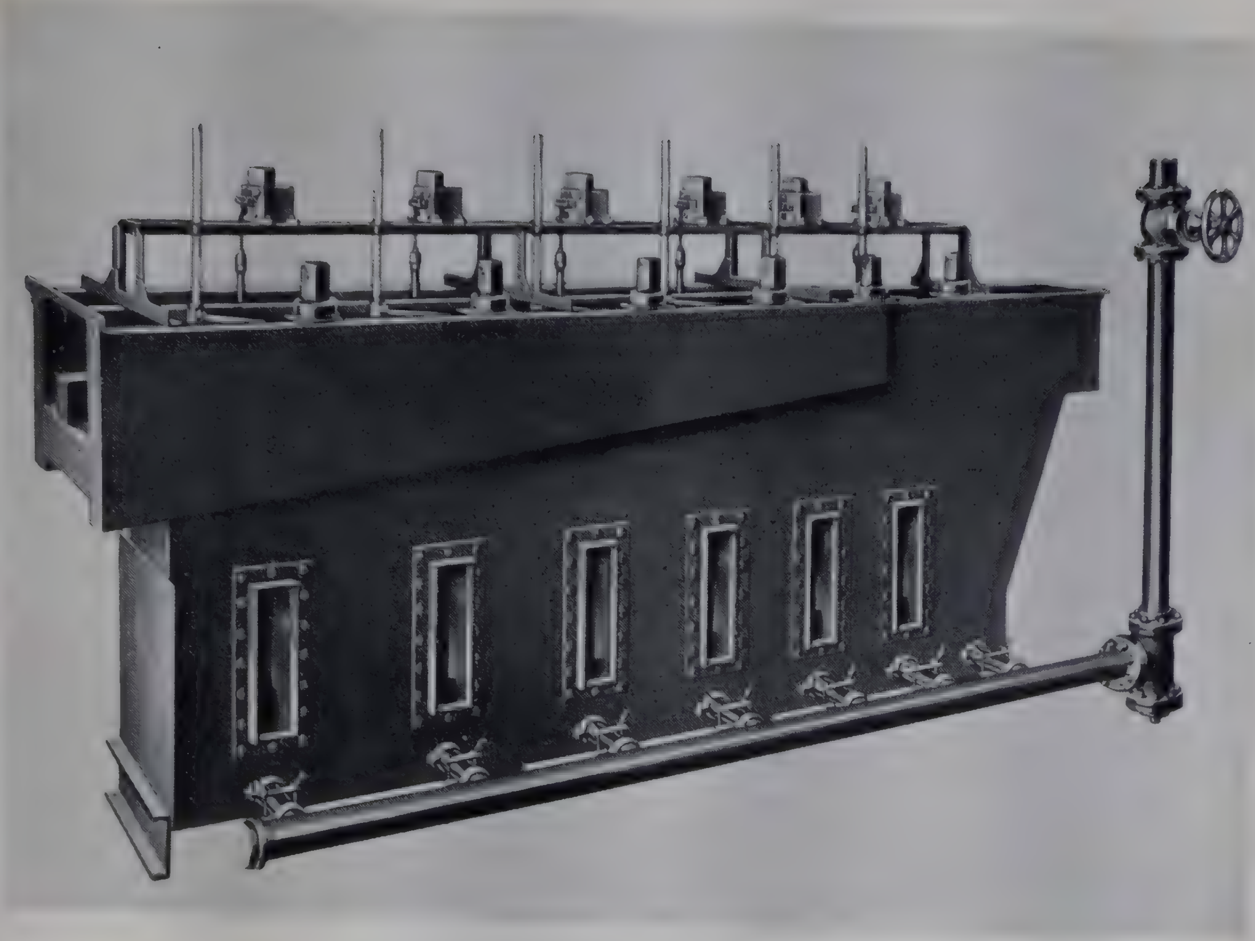

Fig. 25.5. Dorrco sizer

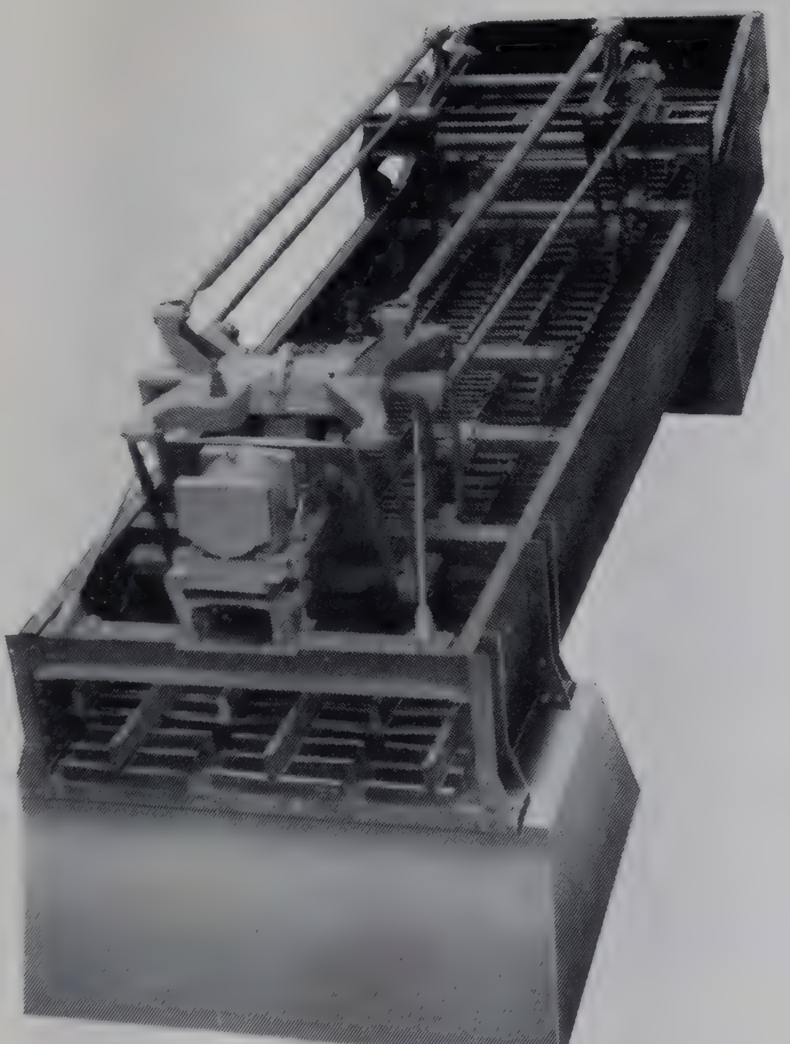

Fig. 25.6. Stokes double-acting rake classifier

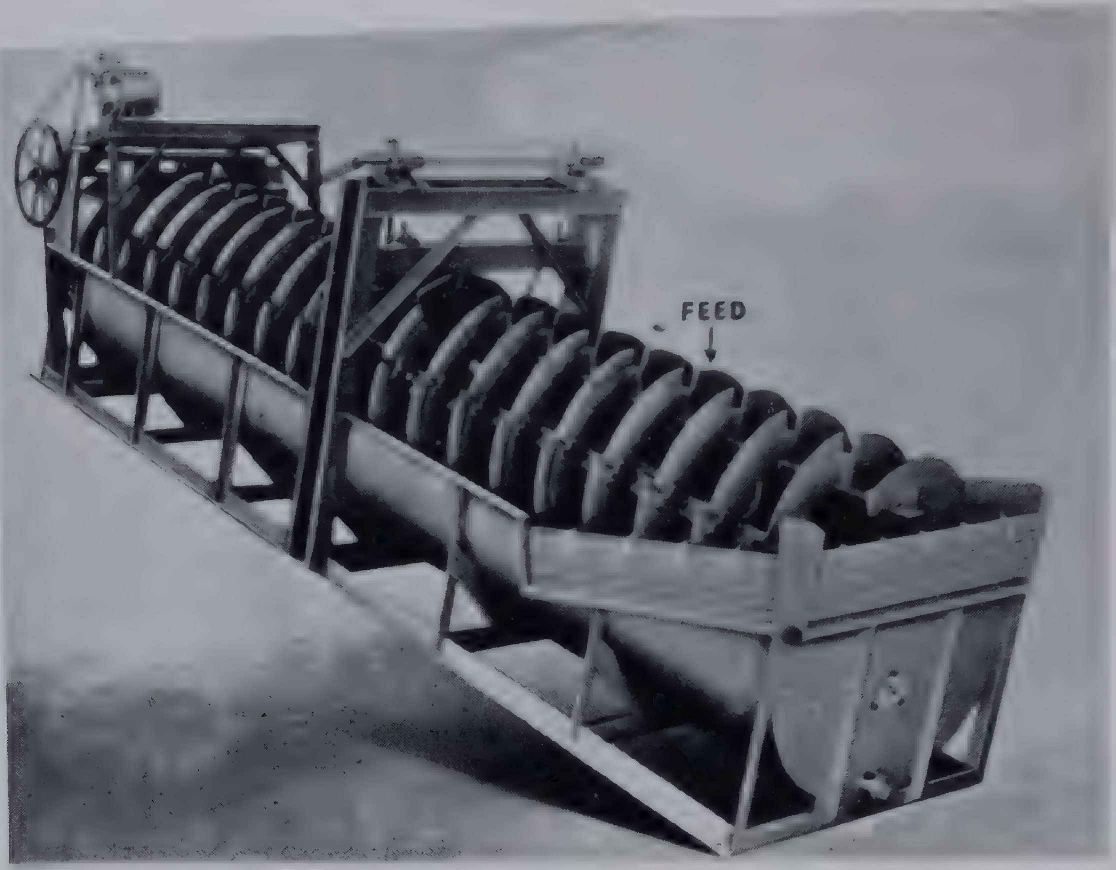

Fig. 25.7. Denver cross-flow classifier

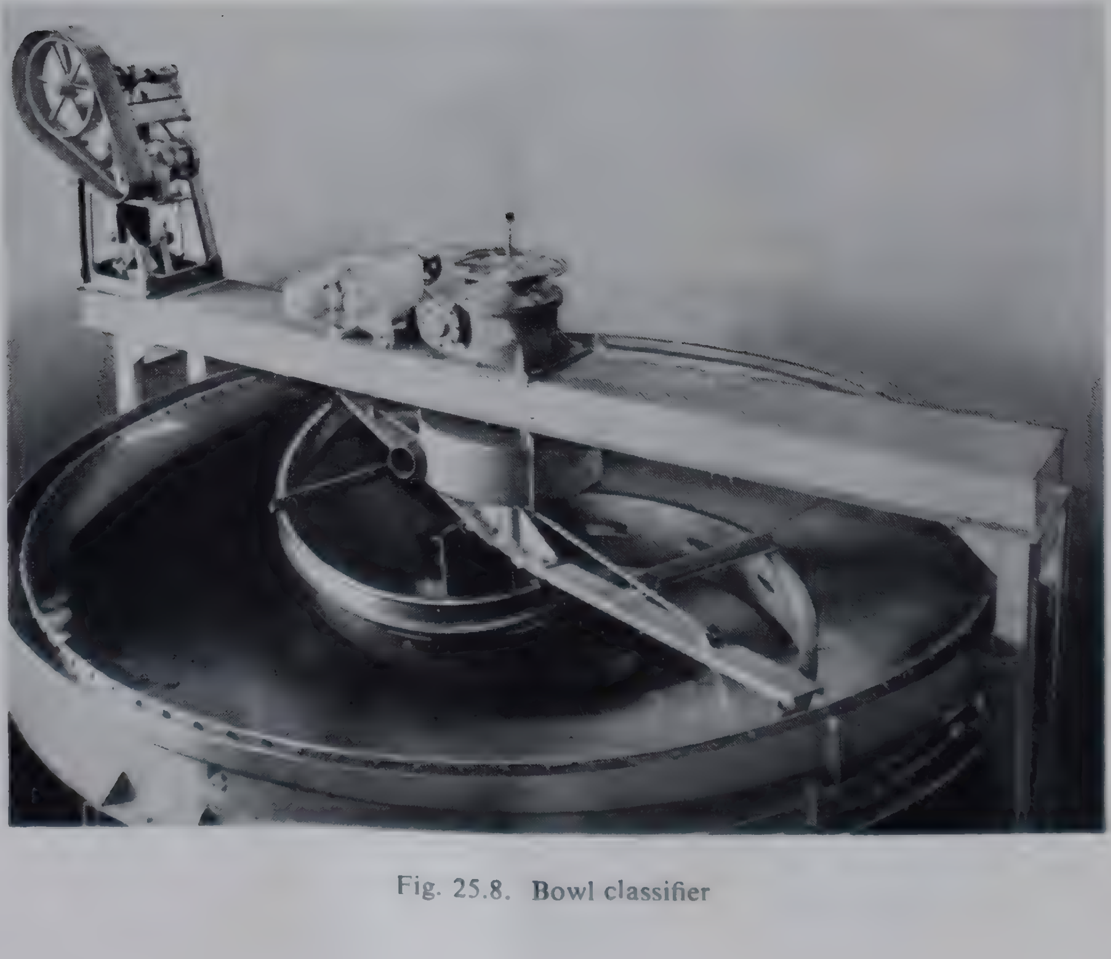

Fig. 25.8. Bowl classifier

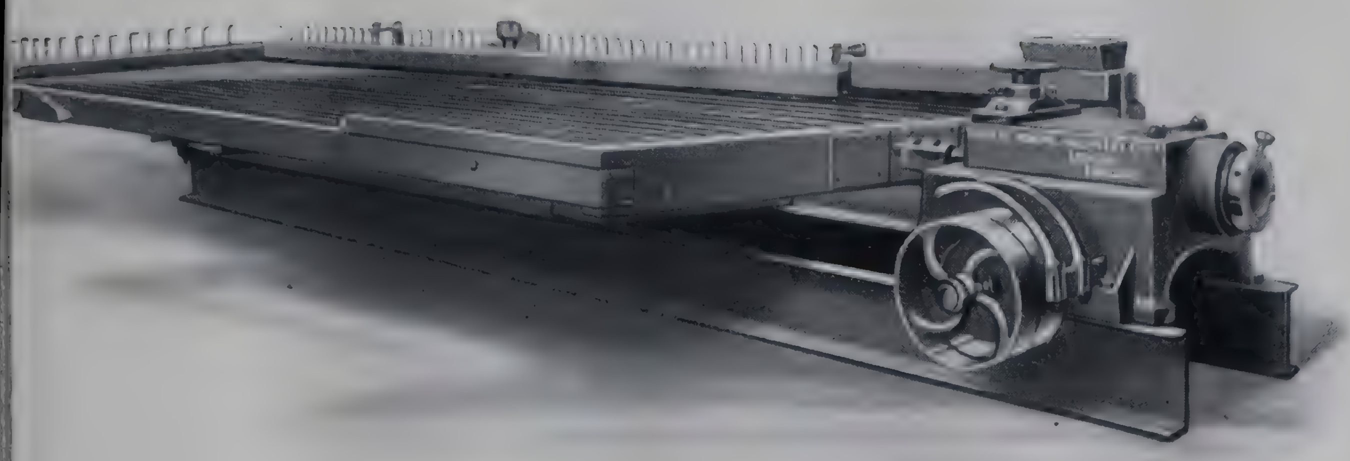

Fig. 25.9. Wilfley riffled table

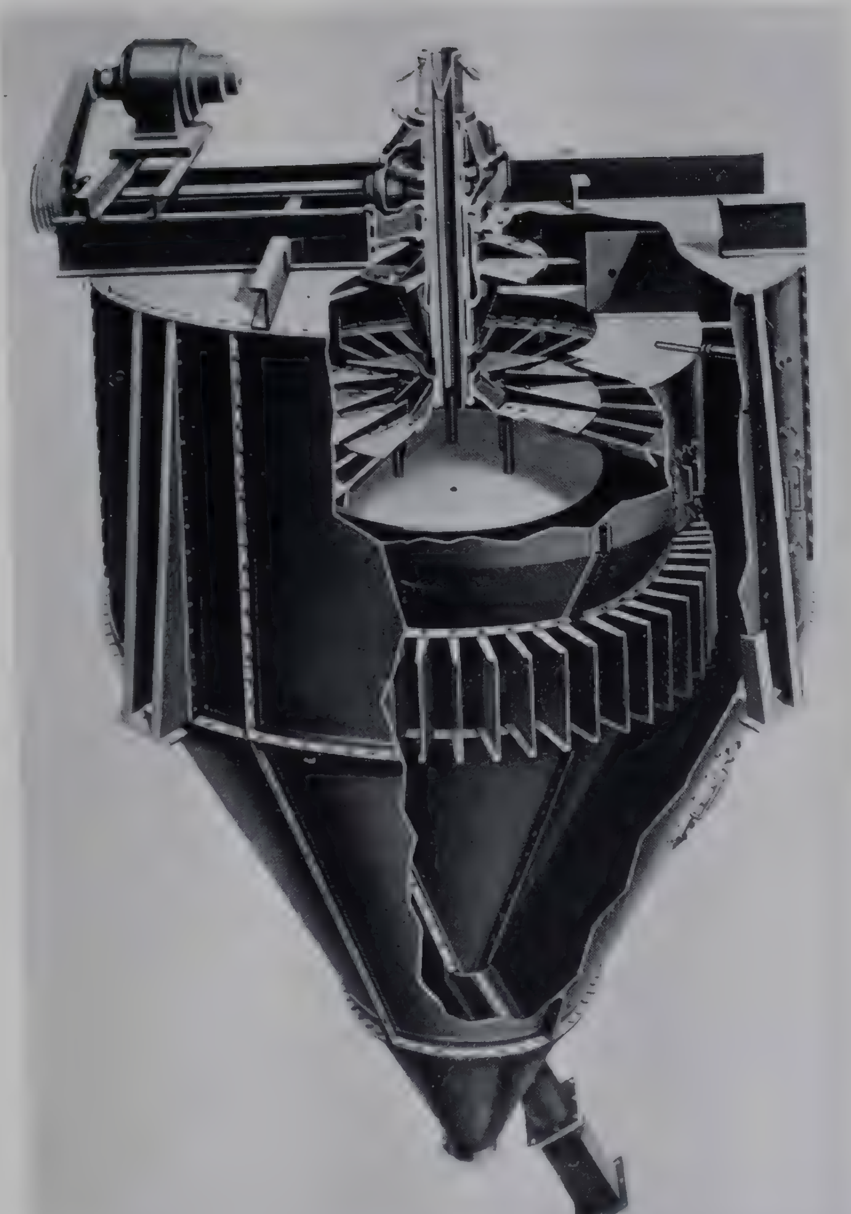

Fig. 25.10. Raymond mechanical air separator

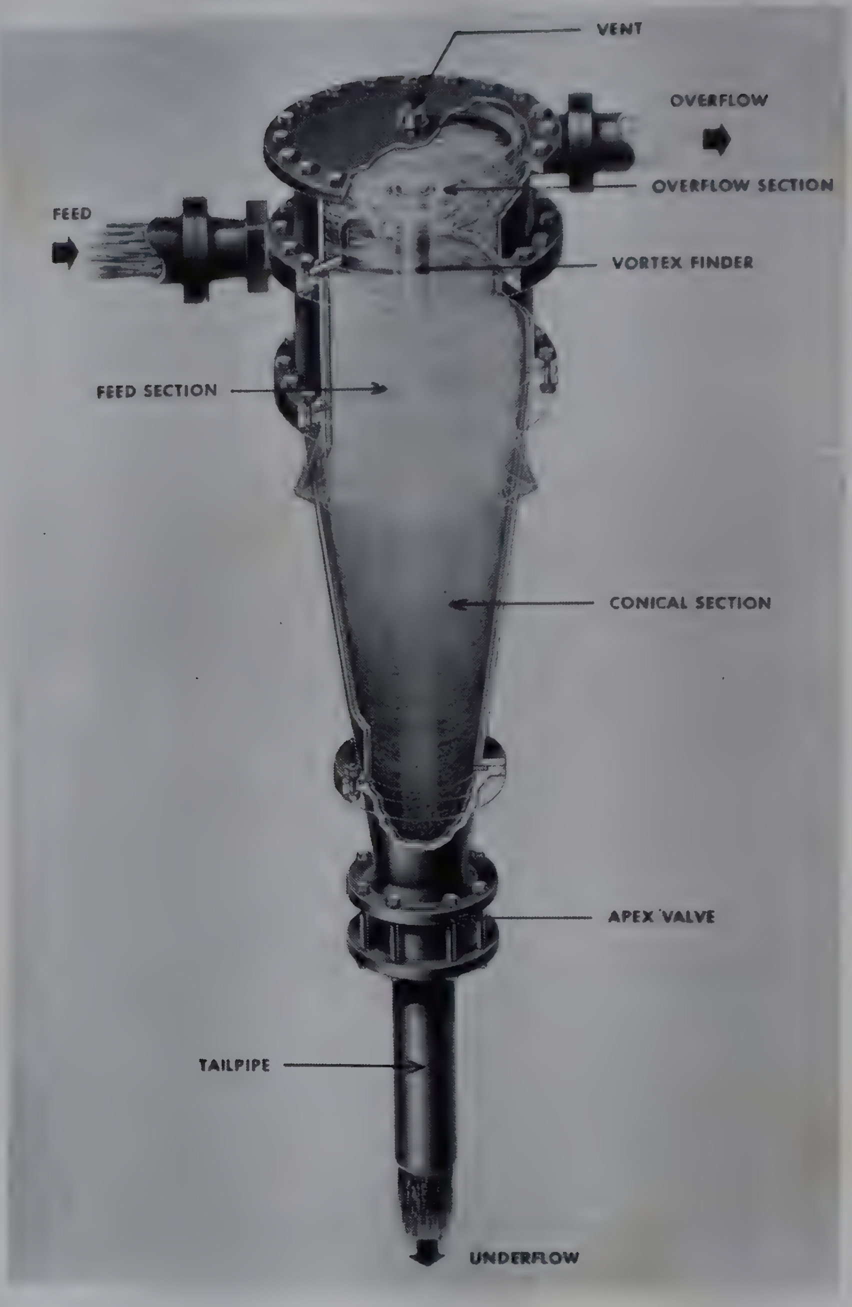
VENT
OVERFLOW
OVERFLOW SECTION
FEED
VORTEX FINDER
FEED SECTION
CONICAL SECTION
APEX VALVE
TAILPIPE
UNDERFLOW

Fig. 25.12. DorrClone centrifugal separator

a rotary motion has been imparted to the air stream, the coarser particles are thrown to the walls of the inner cone and, together with the very large particles, are withdrawn from the bottom. The fine particles remain in suspension and are carried down the space between the two cones and are collected from the bottom of the outer cone. The air returns to the inner cone through a series of deflector vanes, and blows any fines from the surfaces of the larger particles as they fall. The size at which the "cut" is made is controlled by the number of fixed and rotating blades and the speed of rotation.

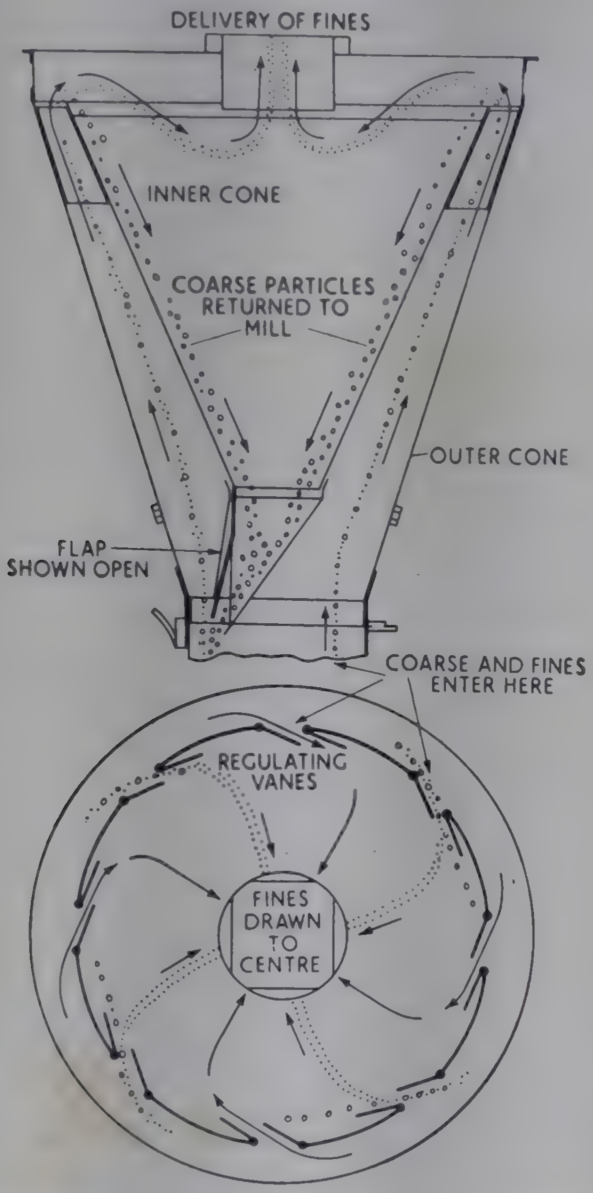
DELIVERY OF FINES
INNER CONE
COARSE PARTICLES RETURNED TO MILL
OUTER CONE
FLAP SHOWN OPEN
COARSE AND FINES ENTER HERE
REGULATING VANES
FINES DRAWN TO CENTRE

Fig. 25.11. Raymond vacuum air separator

Considerable interest has recently been shown in the use of hydraulic cyclone separators and the results of preliminary laboratory investigations have been reported by FERN(17) and by KELSALL(18, 19). The movement of aluminium particles has been studied, and it appears that the flow pattern is similar to that obtained in the cyclone air separator. The main interest in this plant is for the washing of coal, but it is now being used in the metallurgical industry. A commercial model, the DorrClone, is illustrated in Fig. 25.12.

**Sieves or Screens**

Sieves or screens are used industrially on a large scale for the separation of particles according to their sizes, and on the small scale for the production of closely graded materials and for the carrying out of size analyses. The method is applicable for particles of size down to about $\frac{1}{500}$ in., but not for very fine materials because of the difficulty of producing accurately woven fine gauges of sufficient strength and because the screens become clogged. Woven wire cloth is generally used for fine sizes and perforated plates for the larger meshes. Some large industrial screens are formed either from a series of parallel rods or from H-shaped links bolted together, though square or circular openings are more usual.

Even if a particle is sufficiently small, it will pass through the screen only if its configuration relative to the opening is favourable. For a non-isometric particle, therefore, the axis must be correctly aligned with respect to the plane of the screen and the centre of gravity of the particle must be above an aperture. Further, the amount of material on the screen at any instant must be small so that the passage of fine particles is not obstructed by larger ones. The particles should therefore be caused to move vertically as well as horizontally during screening, and a random motion, such as that given by the hand shaking of a small screen, is most satisfactory.

Standard screens always have square apertures, and the size is specified as the length of the side of the square for large sizes, and in terms of the number of openings per inch for the finer screens. There are several standard series of screens; the sizes of the openings are determined by the thickness of wire used. In this country British Standard (B.S.) screens are made in sizes from 300 mesh upwards, but they are too fragile for some work. The Institute of Mining and Metallurgy (I.M.M.) screens are more robust, with the thickness of the wire approximately equal to the size of the apertures. The Tyler series, which is standard in the United States, is intermediate between the two British series. Details of the three series of screens are given in Tables 25.1, 25.2 and 25.3. It is usual to arrange for the apertures of successive screens used for a size analysis to increase by a constant factor of 2, $\sqrt{2}$, or $\sqrt[4]{2}$.

The efficiency of screening is defined as the ratio of the weight of material which passes the screen to that which is capable of passing; this will differ according to the size of the material. It may be assumed that the rate of passage of particles of a given size through the screen is proportional to the number or weight of particles of that size on the screen at any instant. Thus, if $w$ is the weight of particles of a particular size on the screen at a time $t$,

$$\frac{\mathrm{d}w}{\mathrm{d}t} = -kw \qquad \ldots.(25.6)$$

where $k$ is a constant for a given size and shape of particle and for a given screen.

TABLE 25.1. *British Standard Sieves*(4)

| | Nominal meshes per inch | Size of aperture (inches) | Wire diameter or width of bridge (inches) |
|---|---|---|---|
| Perforated plates | | 2 | 0·500 |
| | | $1\frac{7}{8}$ | 0·464 |
| | | $1\frac{3}{4}$ | 0·432 |
| | | $1\frac{5}{8}$ | 0·400 |
| | | $1\frac{1}{2}$ | 0·372 |
| | | $1\frac{3}{8}$ | 0·348 |
| | | $1\frac{1}{4}$ | 0·324 |
| | | $1\frac{1}{8}$ | 0·300 |
| | | 1 | 0·300 |
| | | $\frac{7}{8}$ | 0·276 |
| | | $\frac{3}{4}$ | 0·252 |
| | | $\frac{5}{8}$ | 0·232 |
| | | $\frac{1}{2}$ | 0·192 |
| Wire screens | | $\frac{1}{2}$ | 0·128 |
| | | $\frac{3}{8}$ | 0·104 |
| | | $\frac{1}{4}$ | 0·092 |
| | | $\frac{3}{16}$ | 0·080 |
| | | $\frac{1}{8}$ | 0·072 |
| | | $\frac{1}{16}$ | 0·038 |
| | | $\frac{1}{32}$ | 0·021 |
| | 5 | 0·1320 | 0·068 |
| | 6 | 0·1107 | 0·056 |
| | 7 | 0·0949 | 0·048 |
| | 8 | 0·0810 | 0·044 |
| | 10 | 0·0660 | 0·034 |
| | 12 | 0·0553 | 0·028 |
| | 14 | 0·0474 | 0·024 |
| | 16 | 0·0395 | 0·023 |
| | 18 | 0·0336 | 0·022 |
| | 22 | 0·0275 | 0·018 |
| | 25 | 0·0236 | 0·0164 |
| | 30 | 0·0197 | 0·0136 |
| | 36 | 0·0166 | 0·0112 |
| | 44 | 0·0139 | 0·0088 |
| | 52 | 0·0116 | 0·0076 |
| | 60 | 0·0099 | 0·0068 |
| | 72 | 0·0083 | 0·0056 |
| | 85 | 0·0070 | 0·0048 |
| | 100 | 0·0060 | 0·0040 |
| | 120 | 0·0049 | 0·0034 |
| | 150 | 0·0041 | 0·0026 |
| | 170 | 0·0035 | 0·0024 |
| | 200 | 0·0030 | 0·0020 |
| | 240 | 0·0026 | 0·0016 |
| | 300 | 0·0021 | 0·0012 |

TABLE 25.2. *I.M.M. Screens*(4)

| Meshes per inch | Size of aperture (inches) | Diameter of wire (inches) |
|---|---|---|
| 5 | 0·1 | 0·1 |
| 8 | 0·062 | 0·063 |
| 10 | 0·05 | 0·05 |
| 12 | 0·0416 | 0·0417 |
| 16 | 0·0312 | 0·0313 |
| 20 | 0·025 | 0·025 |
| 30 | 0·0166 | 0·0167 |
| 40 | 0·0125 | 0·0125 |
| 50 | 0·01 | 0·01 |
| 60 | 0·0083 | 0·0083 |
| 70 | 0·0071 | 0·0071 |
| 80 | 0·0062 | 0·0063 |
| 90 | 0·0055 | 0·0055 |
| 100 | 0·005 | 0·005 |
| 120 | 0·0042 | 0·0041 |
| 150 | 0·0033 | 0·0033 |
| 200 | 0·0025 | 0·0025 |

TABLE 25.3. *Tyler Standard Screens*(4)

| Meshes per inch | Size of aperture (inches) | Diameter of wire (inches) |
|---|---|---|
|  | 1·050 | 0·149 |
|  | 0·742 | 0·135 |
|  | 0·525 | 0·105 |
|  | 0·371 | 0·092 |
| 3 | 0·263 | 0·070 |
| 4 | 0·185 | 0·065 |
| 6 | 0·131 | 0·036 |
| 8 | 0·093 | 0·032 |
| 10 | 0·065 | 0·035 |
| 14 | 0·046 | 0·025 |
| 20 | 0·0328 | 0·0172 |
| 28 | 0·0232 | 0·0125 |
| 35 | 0·0164 | 0·0122 |
| 48 | 0·0116 | 0·0092 |
| 65 | 0·0082 | 0·0072 |
| 100 | 0·0058 | 0·0042 |
| 150 | 0·0041 | 0·0026 |
| 200 | 0·0029 | 0·0021 |

Thus, the weight of particles $(w_1 - w_2)$ passing the screen in time $t$ is given by

$$\ln \frac{w_2}{w_1} = -kt$$

$$\therefore \qquad w_2 = w_1 \cdot e^{-kt}. \qquad \ldots .(25.7)$$

If the screen contains a large proportion of material just a little larger than the maximum size of particle which will pass, its capacity is considerably reduced. Screening is generally continued either for a predetermined time or until the rate of screening falls off to a certain fixed value.

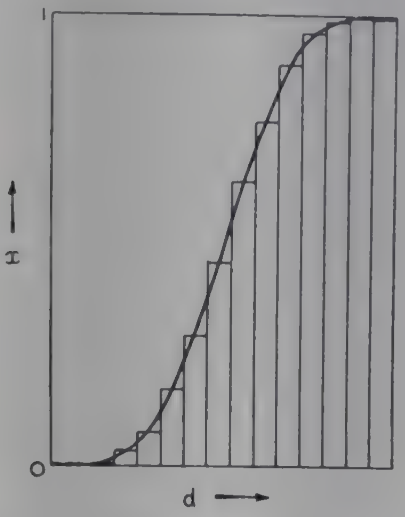

Fig. 25.13. Sieve analysis—cumulative curve

Screening can be carried out either wet or dry. In wet screening, the material is washed evenly over the screen and clogging is prevented; further, the small particles are washed off the surface of the large ones. It has the obvious disadvantage, however, that it may be necessary to dry the material afterwards. With dry screening, the material is sometimes brushed lightly over the screen so as to form a thin even sheet. Care should always be taken to ensure that agitation is not so vigorous that size reduction occurs, because screens are usually quite fragile and easily damaged by rough treatment. In general, the larger and the more abrasive the solids the more robust the screen should be.

The size analysis of a material, as determined by sieving, can be conveniently expressed by plotting the mass fraction of the material which is retained on a given screen against the average size of the particles in the fraction. This average size is usually taken as the arithmetic mean of the apertures of two screens; one through which the particle passes and the other on which the material is retained. This gives a satisfactory method of representing the analyses if the screens are arranged in the form of a regular series. In order to give a better representation of the size analysis for the small particles, a logarithmic scale can be used for the average size. Curves of this type will show a single mode in most cases where the material is all obtained from the same source. If the material consists of a mixture of particles of different origins, then there will, in general, be a number of modes.

The cumulative mass fraction of material below a certain screen size can then be plotted using either linear or logarithmic coordinates, and the points joined to form a smooth curve as shown in Fig. 25.13. This curve will show a region of maximum slope at a size corresponding to the mode on the previous curves. If the slope of this curve is now plotted against particle size, a smooth size frequency curve is obtained for the material, as already indicated in Chapter 24.

***Laboratory Sieves.*** Small screens, known as sieves, for use in the laboratory are usually made from a woven metal cloth which is fixed in a shallow cylindrical framework (Fig. 25.16). The sieves are arranged in sets with the coarsest on top and the finest at the bottom. The material is placed on the top sieve and the set is vibrated either by hand or mechanically (Fig. 25.17). Loss of material is prevented by the use of a tightly fitting lid on the top sieve and a solid tray at the bottom.

***Industrial Screens.*** The only large screen that is hand operated is the grizzly. This has a plane screening surface composed of longitudinal bars up to 10 feet long and fixed in a rectangular framework. In some cases, a reciprocating motion is imparted to alternate bars so as to reduce the risk of clogging. The grizzly is usually inclined at an angle to the horizontal; the greater the angle the greater is the throughput but the lower is the screening efficiency. If the grizzly is used for wet screening, a very much smaller angle is employed. In some screens the longitudinal bars are replaced by a perforated plate.

The mechanically operated screens are vibrated by means of an electromagnetic device (Fig. 25.18), or mechanically (Fig. 25.19). In the former case, the cloth itself is vibrated, and in the latter the whole assembly. Because very rapid accelerations and retardations are produced, the power consumption and the wear on the bearings are high. These screens are sometimes mounted in a multi-deck fashion with the coarsest screen on the top, either horizontally or inclined at angles up to 45°. With the horizontal machine, the vibratory motion fulfills the additional function of moving the particles across the screen.

The screen area which is required for a given operation cannot be predicted without testing the material under similar conditions on the small plant. In particular, the tendency of the material to clog the screening surface can only be determined by experiment.

A very large mechanically operated screen is the trommel (Fig. 25.20), which consists of a slowly rotating perforated cylinder with its axis at a slight angle to the horizontal. The material to be screened is fed in at the top and gradually moves down the screen and passes over apertures of gradually increasing size, with the result that all the material has to pass over the finest screen. There is therefore a tendency for blockage of the apertures by the large material and for oversize particles to be forced through; further, the relatively fragile fine screen is subjected to the abrasive action of the large particles. These difficulties are obviated to some extent by arranging the screens in the form of concentric cylinders with the coarsest in the centre. The disadvantage of all screens of this type is that only a small fraction of the screening area is in use at any one time. The speed of rotation of the trommel should not be high enough for the material to be carried completely round in contact with the screening surface. The lowest speed at which this occurs is known as the critical speed and is analogous to the critical speed of the ball mill; speeds of between one third and a half of the critical speed are usually recommended. In a modified form of the trommel, the screen surfaces are in the form of truncated cones. Such screens are mounted with their axes horizontal and the material flows away from the apex of the cone.

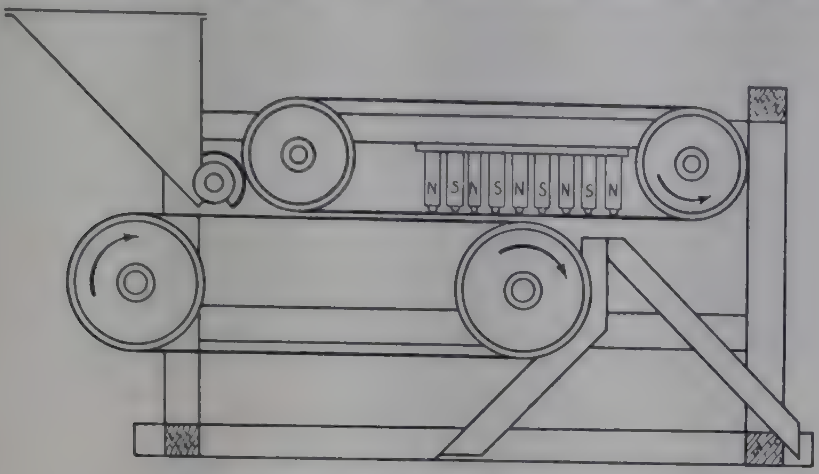

Fig. 25.14. Ball-Norton magnetic separator

### Magnetic Separators

In the magnetic separator, material is passed through the field of an electromagnet which causes the retention or retardation of the magnetic constituent, It is important that the material should be supplied as a thin sheet in order that all the particles are subjected to a field of the same intensity and so that the free movement of individual particles is not impeded. The two main types of equipment are as follows:

1. Eliminators are used for the removal of small quantities of magnetic material from the charge to a plant. They are frequently employed, for example, for the removal of stray pieces of scrap iron from the feed to crushing equipment. A common type of eliminator is a magnetic pulley (Fig. 25.21) incorporated in a belt conveyor so that the non-magnetic material is discharged in the normal manner but the magnetic material adheres to the belt and falls off from the underside.

2. Concentrators are used for the separation of magnetic ores from the accompanying mineral matter. The Ball-Norton machine (Fig. 25.14), which is a typical concentrator, employs two horizontally staggered belt conveyors running parallel, one above the other. The material is fed as a thin sheet to the lower belt and is conveyed under the second belt where it is subjected to the action of a magnetic field. The non-magnetic material is discharged in the normal manner, but the magnetic material adheres to the lower side of the upper belt. The time for which the material is subjected to the magnetic field can be varied by modifying the speed of the belts or by altering the overlap.

Another concentrator is the Davies magnetic separator (Fig. 25.22) in which material is fed in a thin sheet over a magnetic feed plate and the magnetic constituent is picked up by rotating electromagnets. The current is automatically switched off as the electromagnets pass over a collecting bin into which the magnetic material then falls. The non-magnetic constituent passes directly over the feed plate into a collecting hopper.

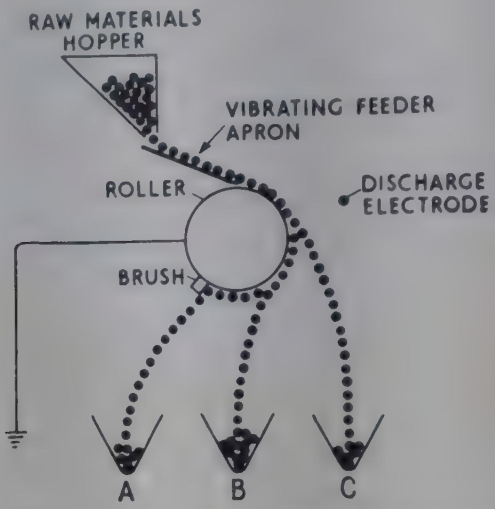

Fig. 25.15. Electrostatic separator

### Electrostatic Separators

Electrostatic separators, in which differences in the electrical properties of the materials are exploited in order to effect a separation, are now sometimes used with small quantities of fine material. The solids are fed from a hopper on to a rotating drum, which is either charged or earthed, and an electrode bearing the opposite charge is situated at a small distance from the drum (Fig. 25.15). The point at which the material leaves the drum is determined by the charge it acquires, and by suitable arrangement of the collecting bins (*A*, *B*, *C*) a sharp classification can be obtained.

### Froth Flotation

Separation of a mixture using flotation methods depends on differences in the surface properties of the materials involved. If the mixture is suspended in an aerated liquid, the gas bubbles will tend to adhere preferentially to one of the constituents—the one which is more difficult to wet by the liquid—and its effective density may be reduced to such an extent that it will rise to the surface. If a suitable frothing agent is added to the liquid, the particles will be held in the surface by means of the froth until they can be discharged over a weir. Froth flotation is widely used in the metallurgical industries where, generally, the ore is difficult to wet and the residual earth is readily wetted.

The process depends on the existence, or development, of a selective affinity of one of the constituents for the envelopes of the gas bubbles. In general, this affinity must be induced, and the reagents which increase the angle of contact between the liquid and one of the materials are known as promoters and collectors. Promoters are selectively adsorbed on the surface of one material

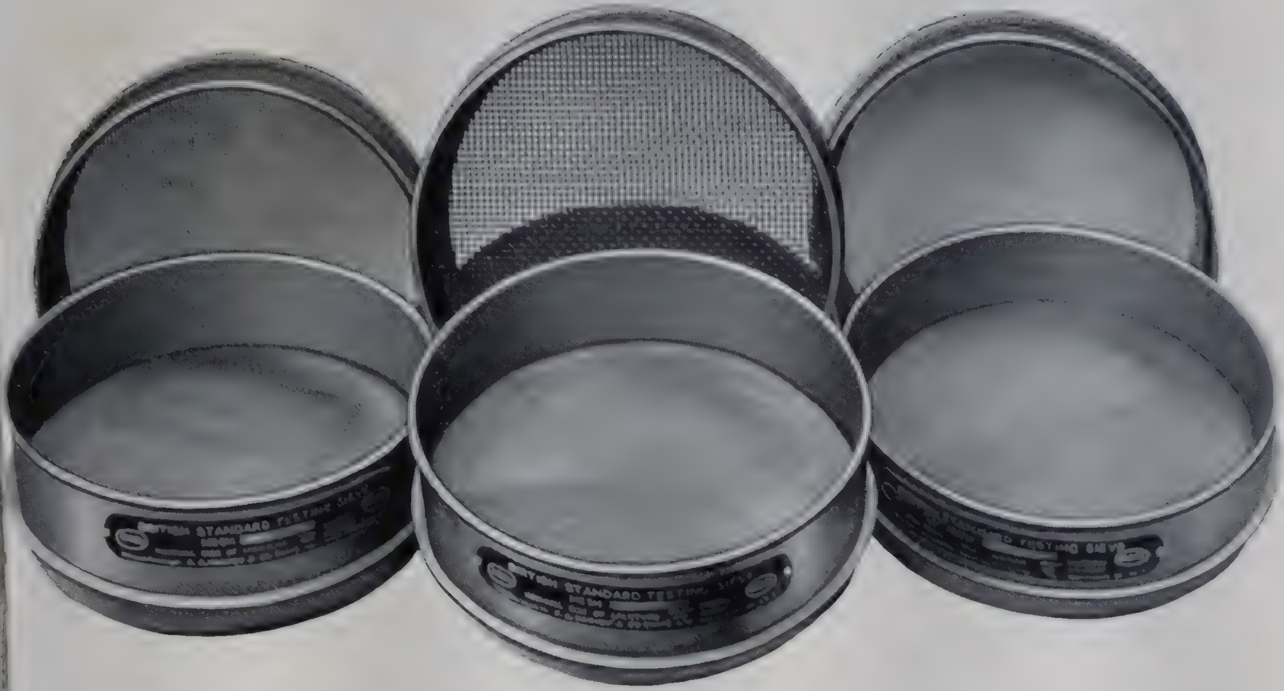

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Fig. 25.16. Laboratory screens

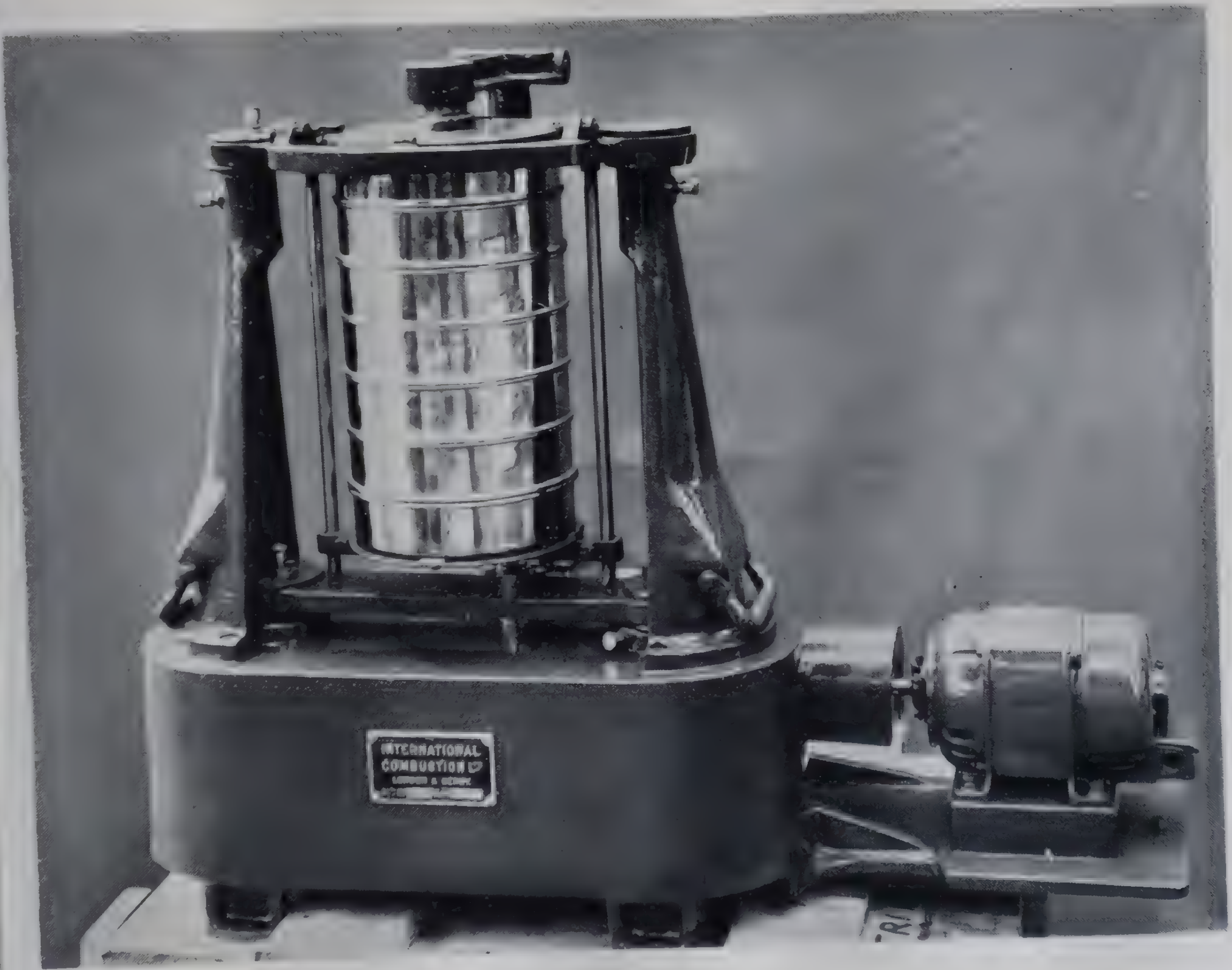

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Fig. 25.17. Rotap sieve shaker

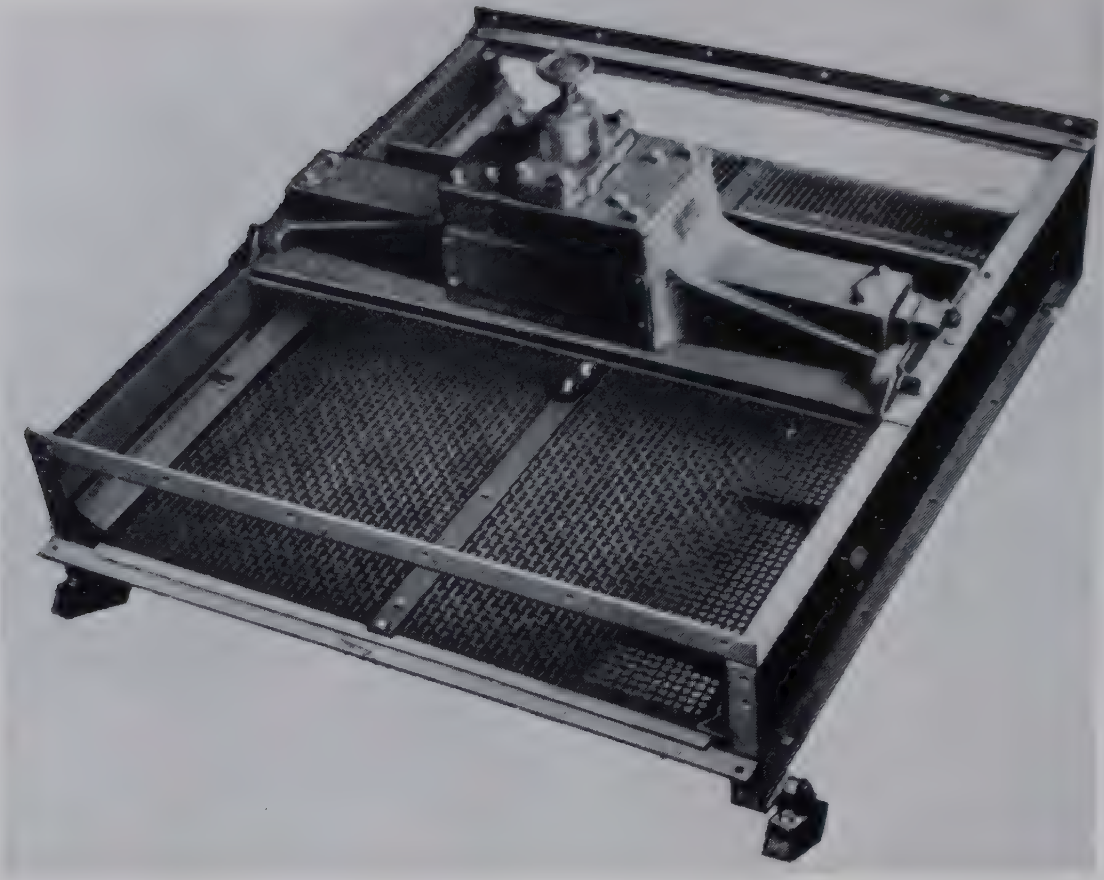


Fig. 25.18. Hummer electromagnetic screen

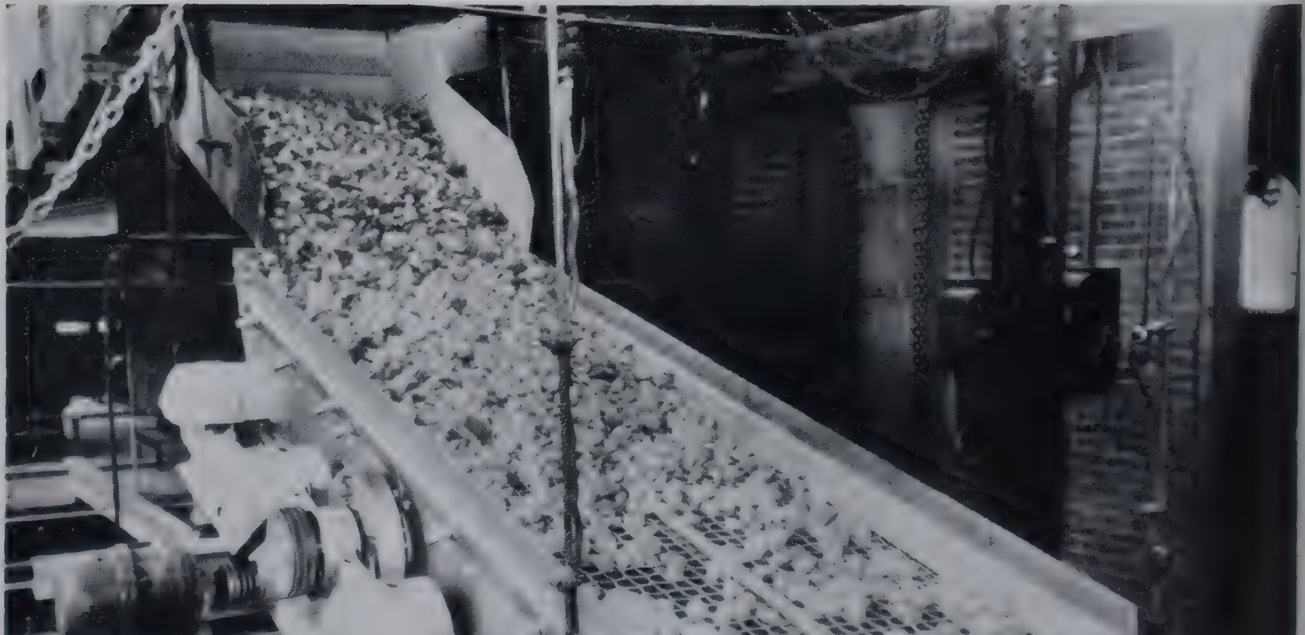


Fig. 25.19. Tyrock mechanical screen

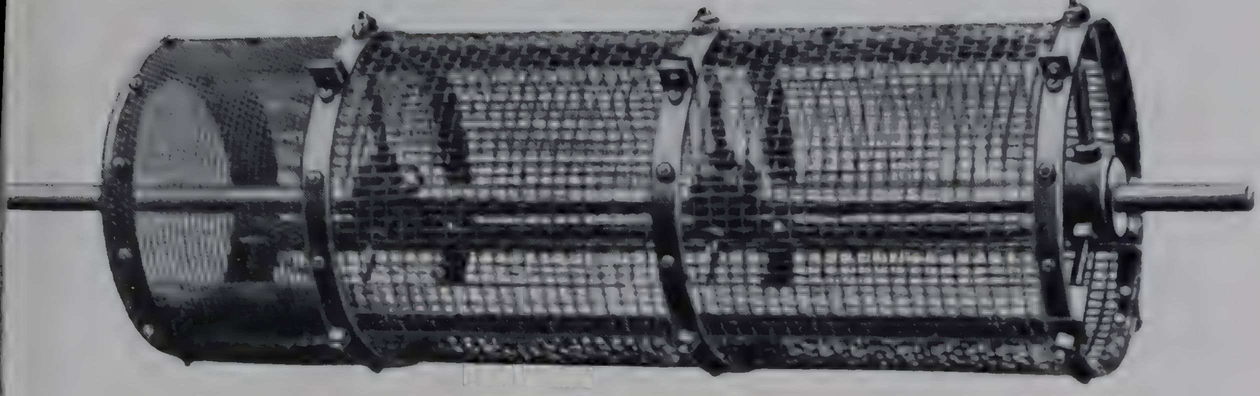


Fig. 25.20. Trommel

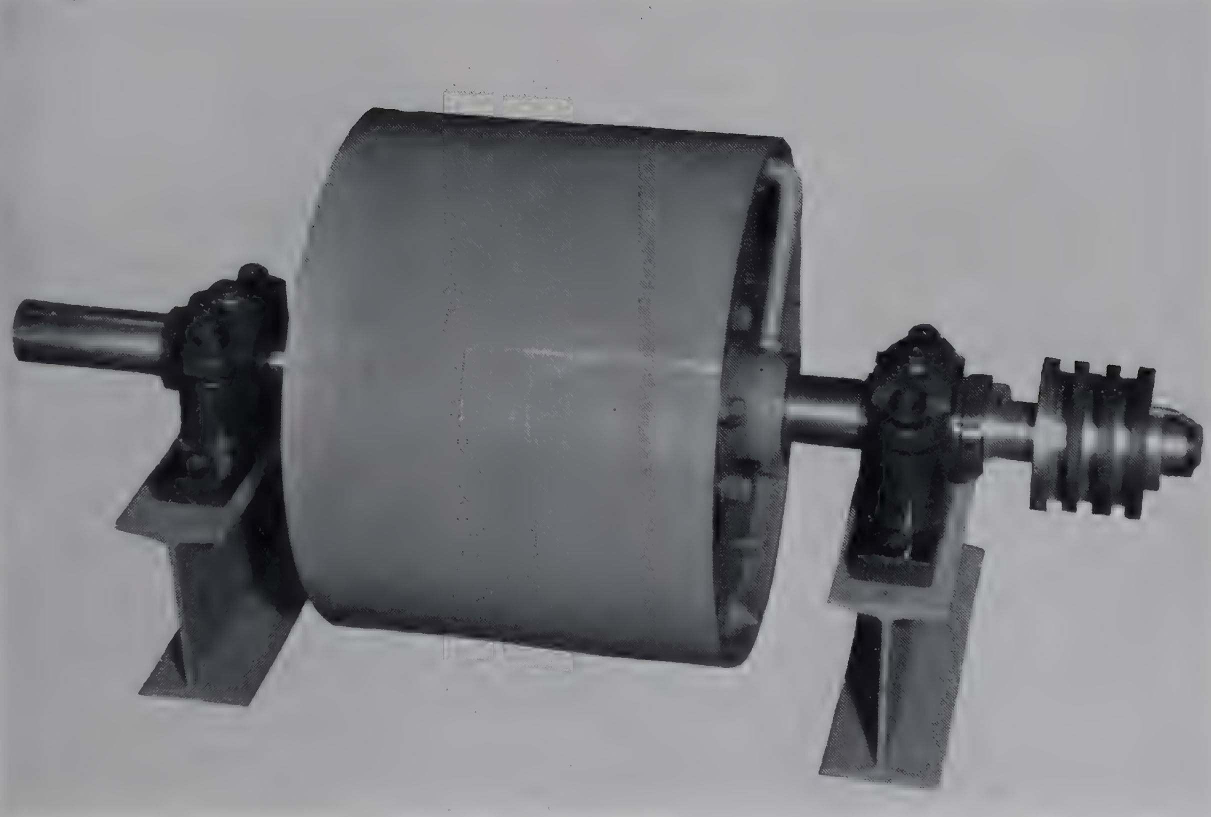
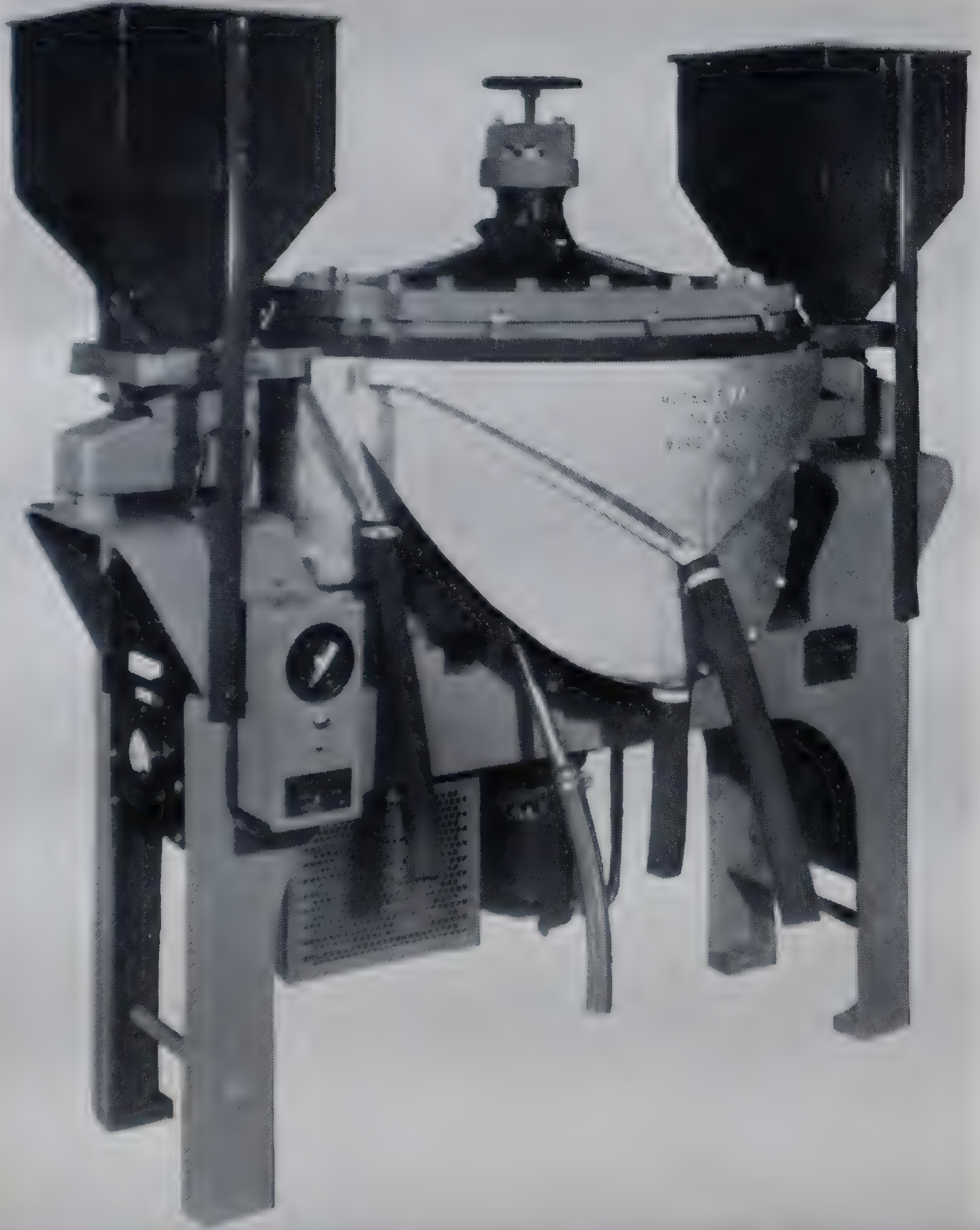
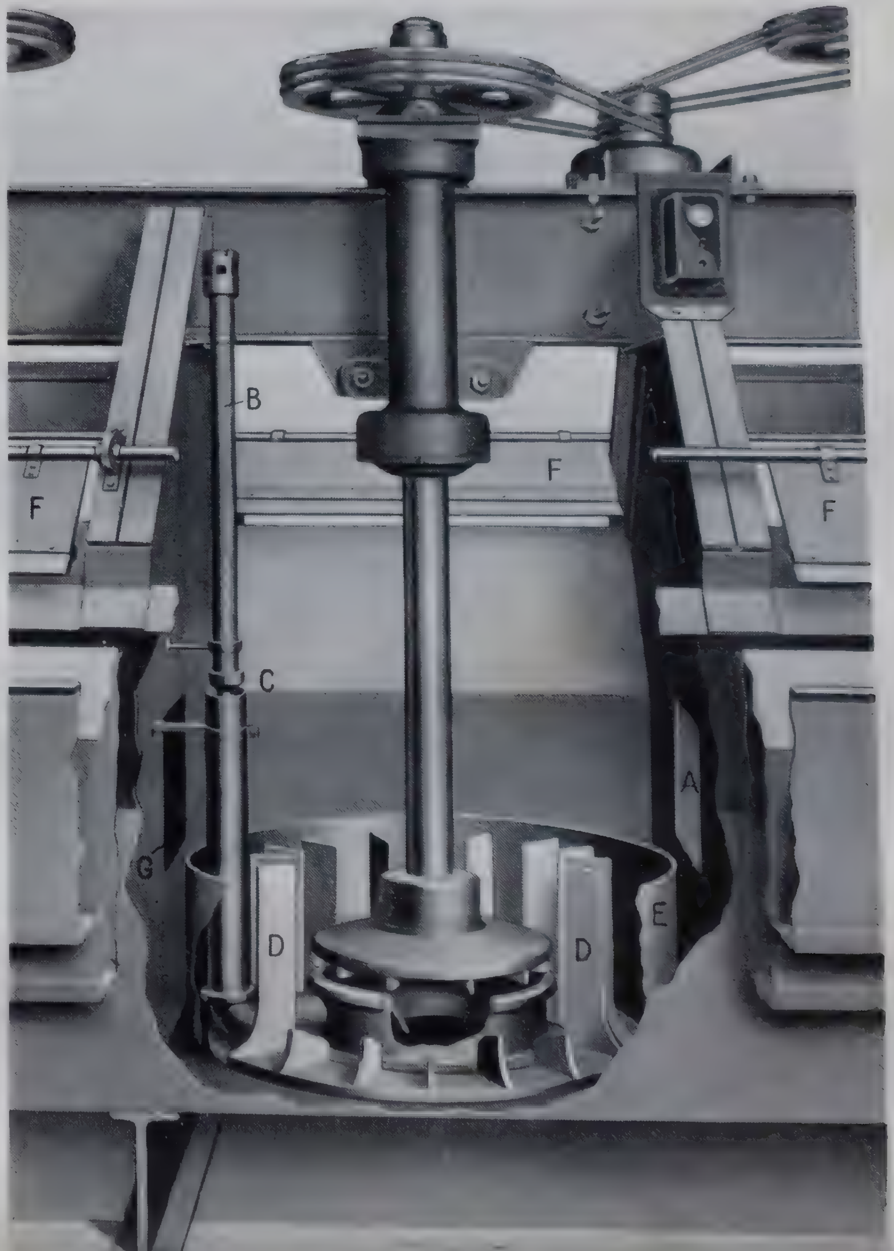


Fig. 25.21. Magnetic pulley




B
F
F
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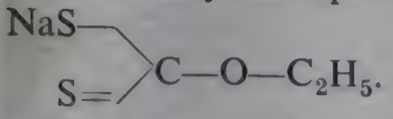
D

and form a monomolecular layer; use of excess material destroys the effect. Concentrations of the order of 0.1 pound per ton of solids are usually required. A commonly used promoter is sodium ethyl xanthate which has the formula



$$\begin{matrix} NaS- \\ S= \end{matrix} \!\!> C-O-C_2H_5$$

In solution this material ionises giving positively charged sodium ions and negatively charged xanthate ions. The polar end of the xanthate ion is adsorbed and the new surface of the particles is therefore made up of the non-polar part of the radicle, so that the contact angle with water is increased. A large number of other materials are used in particular cases; for instance, other xanthates and diazoaminobenzene. Collectors are materials which form surface films on the particles; these films are rather thicker than those produced by promoters, and collectors therefore have to be added in higher concentrations—about 1 pound per ton of solids. Pine oil is a commonly used collector, and petroleum compounds are frequently used though they often form very greasy froth which is then difficult to disperse.

In many cases, it is necessary to modify the surface of the other constituent so that it does not adsorb the collector or promoter. This is effected by means of materials known as modifiers which are either adsorbed on the surface of the particles or react chemically at the surface, and thereby prevent the adsorption of the collector or promoter. Mineral acids, alkalies and salts are frequently used for this purpose.

An essential requirement of the process is the production of a froth of sufficient stability to retain the particles of the one constituent in the surface so that they can be discharged over the overflow weir. On the other hand, the froth should not be so stable that it is difficult to break down at a later stage. The frothing agent should reduce the interfacial tension between the liquid and gas, and the quantities required are less when the frothing agent as adsorbed at the interface. Liquid soaps, soluble oils, cresol and alcohols, in the range between amyl and octyl alcohols, are frequently used as frothing agents. In many cases, the stability of the foam is increased by adding a stabiliser—usually a mineral oil—which increases the viscosity of the liquid film forming the bubble, and therefore reduces the rate at which liquid can drain from the envelope. Pine oil is sometimes used as a frothing agent; it produces a stable froth and acts as a collector, in addition.

The reagents which are used in froth flotation are usually specific in their action and the choice of suitable reagents is usually made as a result of tests on small scale equipment. Mixtures of more than two components can be separated in stages using different reagents at each stage. Sometimes the behaviour of a system is considerably influenced by change in the $p_H$ of the solution, and it is then possible to remove materials at successive stages by progressive alterations in the $p_H$. In general, froth flotation processes can be used for particles between about 5 microns and 65 mesh in size. The tendency of various materials to respond to froth flotation methods is given by DOUGHTY(11).

***The Callow Cell.*** The Callow cell (Fig. 25.24) is a typical froth flotation unit. It consists of a tank with an inclined bottom covered with a porous material through which air is continuously blown. A suspension of the material is prepared and the necessary chemicals are added; it is fed in to the shallow end of the tank. The suspension flows under a weir and is thus brought to the bottom of the tank where it is subjected to the action of the compressed air which is introduced at a number of points below the porous medium. This method of introducing air is necessary in order to obtain an even distribution in a tank in which the hydrostatic head varies. In some cases, the mixture of solid, reagent and liquid is made up in a conditioning tank where the material acquires its surface film before being introduced into the cell.

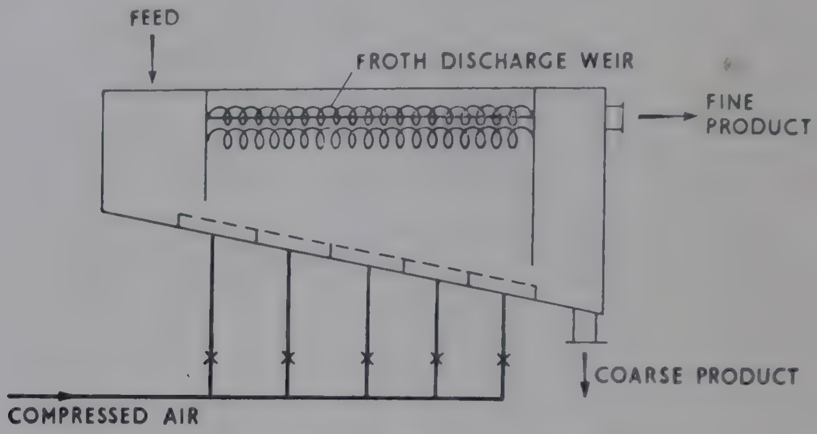


Fig. 25.24. Simple Callow cell

The wetted constituent is removed at the lower end of the tank and is easily separated from the liquid. The other material is carried over a weir in the froth and is either allowed to settle in a thickener or is fed to a rotary filter for the recovery of the solid. Either fraction from the cell may then pass on to a second unit if inadequate separation has been obtained or if the mixture consists of more than two components.

The simple Callow cell is rarely used now however because the air inlets in the porous medium tend to become obstructed, especially when mixtures of high concentrations are separated, and high pressures are necessary in order to maintain the required air flow. Various modifications of the Callow cell are now used. In one model the air is introduced through a slowly rotating perforated metal cylinder covered with a porous cloth; the solids do not then obstruct the air flow because they are thrown off as the cylinder rotates. In another model the air is introduced in a series of vertical pipes terminating in air distributors at the bottom of the tank. Occasionally, these cells are operated under reduced pressure because the density of the gas is thereby reduced and an increased lift is obtained.

A modern froth flotation cell used for coal washing is illustrated in Fig. 25.23. The suspension, containing about 20% of solids together with the necessary reagents, enters at *A*, and the rotating impeller draws in air through the pipe *B*, together with some suspension. The air volume is controlled by a valve at the inlet, and the volume of suspension by an adjustable cone at *C*. The aerated suspension passes through the baffles *D* which destroy the vortex, and then is thrown against a rubber-covered wear ring *E*. The froth rises to the surface and is driven over the froth weirs by means of rotating paddles *F*. The rejects are discharged through the port *G*, either to waste or to a further unit. In the latter case, the flow is maintained by allowing a small fall in liquor level from cell to cell.

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## LIST OF SYMBOLS USED IN CHAPTERS 24 AND 25

| | | |
|---|---|---|
| $b$ | Half distance between crushing rolls | **L** |
| $C$ | A coefficient | $\mathbf{L}^{1-p}\mathbf{T}^{-2}$ |
| $c$ | Radius of yoke of Raymond mill | **L** |
| $d$ | A characteristic linear dimension | **L** |
| $d_A$, $d_B$ | Equivalent spherical diameters of particles of *A* and *B* | **L** |
| $d_l$ | Linear mean diameter | **L** |

| | | |
|---|---|---|
| $d_l'$ | Mean linear diameter | L |
| $d_s$ | Surface mean diameter | L |
| $d_s'$ | Mean surface diameter | L |
| $d_v$ | Volume mean diameter | L |
| $d_v'$ | Mean volume diameter | L |
| $E$ | Energy per unit mass | $L^2T^{-2}$ |
| $E_i$ | Work index | $L^2T^{-2}$ |
| $f_c$ | Crushing strength of material | $ML^{-1}T^{-2}$ |
| $g$ | Acceleration due to gravity | $LT^{-2}$ |
| $K_K$ | Kick's constant | $M^{-1}L^3$ |
| $K_R$ | Rittinger's constant | $M^{-1}L^4$ |
| $k$ | Constant for given size and shape of particle and given screen | $T^{-1}$ |
| $k_1$ | Constant relating to volume of particle | — |
| $k_2$ | Constant relating to surface of particle | — |
| $L$ | Characteristic linear dimension | L |
| $l$ | Length of arm of Raymond mill | L |
| $M$ | Mass of crushing head in Raymond mill | M |
| $M'$ | Mass of arm of Raymond mill | M |
| $M''$ | Total mass of particles | M |
| $m$ | Mass per unit length of arm of Raymond mill | $ML^{-1}$ |
| $n$ | Number of particles of size $d$ in unit mass of material | $M^{-1}$ |
| $p$ | A constant used as an index | — |
| $q$ | Size reduction $L_1/L_2$ | — |
| $r$ | Radius of ball mill less radius of particle | L |
| $r_1$ | Radius of crushing rolls | L |
| $r_2$ | Radius of particle in feed | L |
| $R$ | Normal reaction | $MLT^{-2}$ |
| $S$ | Surface of particles per unit mass of total material | $M^{-1}L^2$ |
| $s$ | An index | — |
| $t$ | Time | T |
| $u_{0A}$ | Terminal falling velocity of particle of material $A$ | $LT^{-1}$ |
| $u_{0B}$ | Terminal falling velocity of particle of material $B$ | $LT^{-1}$ |
| $w$ | Weight of particles on screen at time $t$ | $MLT^{-2}$ |
| $w_1 - w_2$ | Weight of particles passing through screen in time $t$ | $MLT^{-2}$ |
| $x$ | Mass fraction of particles of size $d$ | — |
| $y$ | Distance along arm from point of support | L |
| $\alpha$ | Half angle of nip | — |
| $\mu$ | Viscosity of fluid | $ML^{-1}T^{-1}$ |
| $\rho$ | Density of fluid | $ML^{-3}$ |
| $\rho_A$ | Density of material $A$ | $ML^{-3}$ |
| $\rho_B$ | Density of material $B$ | $ML^{-3}$ |
| $\rho_s$ | Density of solid material | $ML^{-3}$ |
| $\theta$ | Angle between axis and vertical | — |
| $\omega$ | Angular velocity | $T^{-1}$ |
| $\omega_c$ | Critical speed of rotation of ball mill | $T^{-1}$ |

## CHAPTER 26

# *Mixing and Agitation*

THE problem of mixing two or more substances has proved one of the most intractable of all the unit operations of chemical engineering, and the vast majority of industrial equipment is still designed from experience rather than from any agreed fundamental theory. There is at present no theoretical standard by which the performance of a mixer can be judged, and the assessment of the suitability of a given piece of equipment is always a comparative one. Mixing is usually carried out for one of the following reasons:—

1. To promote intimate contact between the materials, and consequently to provide better control of a chemical reaction. Thus, in nitration, good agitation prevents excessive localised reaction with the formation of undesirable products. As we have seen, agitation is an important feature in crystallisers, dryers, thickeners and other equipment, but in reaction vessels adequate agitation is essential if the desired products are to be obtained.

2. To provide materials of new properties not necessarily present in the ingredients. Thus, gunpowder is a mixture of carbon, sulphur, and potassium nitrate, yet has quite distinct properties. Good mixing of paints avoids the formation of streaks on application, and can result in new tints. The preparations of mixed fertilisers, and of many pharmaceutical products, are common examples of processes demanding control of the mixing process.

### Important Properties of Materials

Mixing may be effected between any two phases, but the problems of liquid-liquid mixing, the dispersion of solids in fluids, and the mixing of solids are the main items in this chapter. The more important properties of the materials that may influence the ease of mixing are:—

*Fluids.* Viscosity, density, relative density, and miscibility.

*Solids.* Fineness, density, relative density, shape, stickiness, and wettability.

The relative proportions and the number of components will considerably influence the rate of mixing. The consistency or apparent viscosity will influence the type of flow pattern obtained in a two-phase system with a given type of stirrer. In Fig. 26.1, the rate of flow is plotted against the stress: with Newtonian fluids (*A*) the rate of flow produced by a stirrer is a linear function of the stress, and however small the stress some flow will result. For dense suspensions or other media which show plasticity (*C*), a minimum stress is required before any flow occurs. With pseudoplastic substances (*B*) the slope of the curve is very small at low stresses but it increases rapidly at higher stresses; these materials differ from those of type *C* in that the curve does pass through the origin. Curves of this type can be obtained using a Stormer viscometer, where the force required to rotate a stirrer of standard dimensions is taken as a measure of the stress. Typical curves$^{(1)}$ are shown in Fig. 26.2.

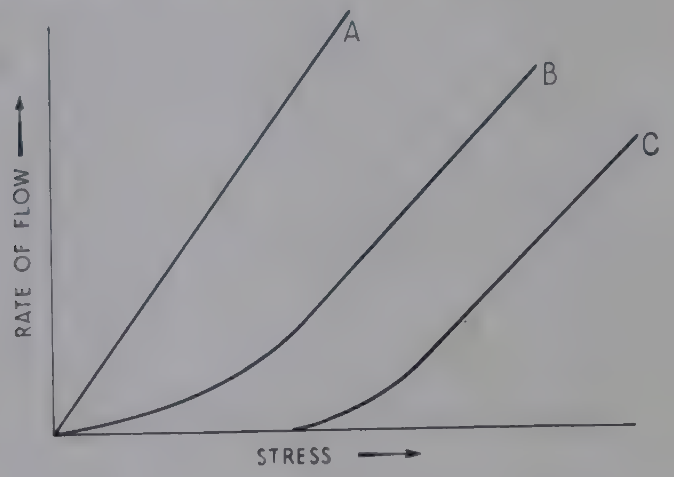


Fig. 26.1. Typical consistency curves
*A* Newtonian fluid. *B* Pseudoplastic material *C* Plastic material

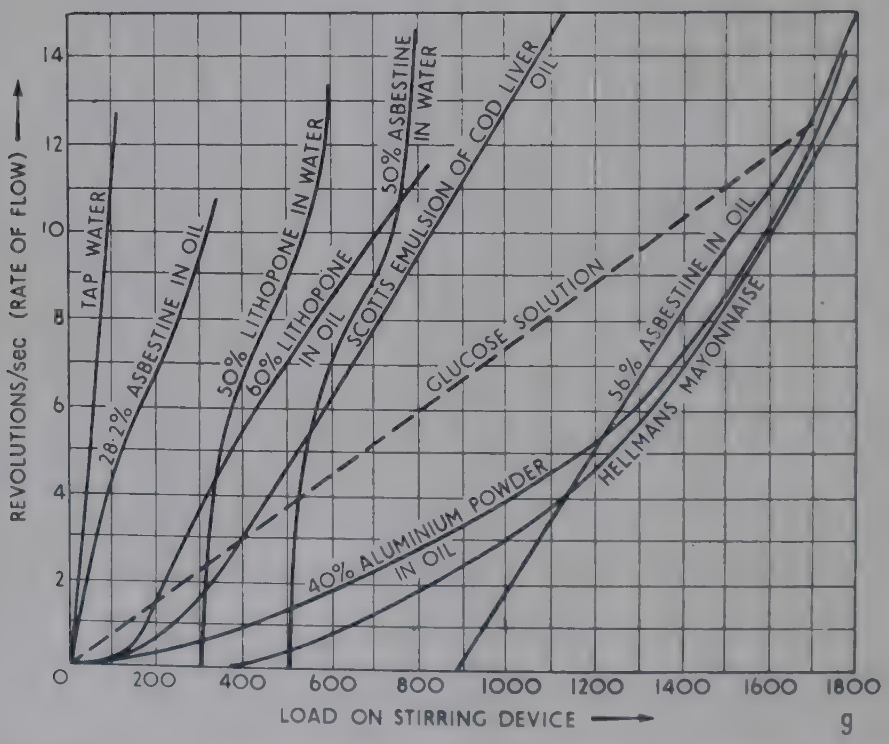


Fig. 26.2. Rate of shear in a Stormer viscometer as a function of load

## Equipment

Mixing equipment may be designed for batch or continuous operation. On the whole, small units give a higher degree of dispersion, and large equipment is in general used only for the agitation of mobile liquids. Whilst gases and thin liquids or suspensions can be handled in continuous mixers, very viscous or plastic materials must usually be treated in a batch system. Continuous mixers are represented by simple jets for gases, centrifugal pumps for liquids and belt conveyors used as simple blenders for solids. Simple cylindrical vessels with agitators may be used either as batch or as continuous mixers for liquids and suspensions, according to the quantity to be handled and the physical properties of the materials. The series of kettles forming a multistage reactor in the nitration of toluene or the sulphonation of benzene is an example of this arrangement. When the equipment is used as a batch mixer, the following points are of importance:

(1) Production of the desired results, in a reasonable time,

(2) Ease and quickness of discharge, possibility of cleaning, adaptability, strength of bearings, and

(3) The power consumption.

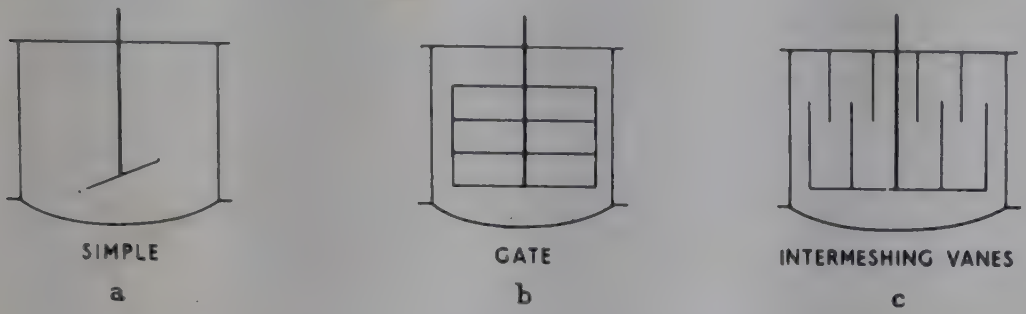


Fig. 26.3. Types of paddle agitators

***Types of Equipment.*** A number of the common types of equipment are illustrated in Figs. 26.3 onwards.

(*A*) *Paddles* (Fig. 26.3). The simplest form of agitator is the straight arm paddle which is commonly used for very simple operations. The gate form is more complex and will handle thicker pastes. With both types, care must be taken to see that the paddle is arranged to give a vertical uplift to the suspension. The addition of intermeshing vanes:

(1) Prevents swirling of the entire mass with thin liquids,

(2) Tends to direct currents at right angles to the blades,

(3) Produces shear with heavy fluids, and

(4) Considerably increases the power consumption.

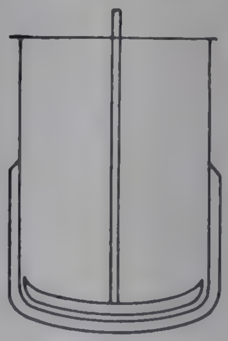


Fig. 26.4. Anchor paddle

(*B*) *Anchor Paddle* (Fig. 26.4). The anchor-type paddle is used in enamel-lined kettles, and can be arranged with a small clearance at the walls. Some form of fixed baffle should be added to prevent vortex formation. This type of unit is frequently used with thick pastes as in the sulphonation of many organic materials, where a propeller working inside a coil (see Fig. 26.19) is unsuitable because of the high viscosity. The scraping action of the agitation dislodges pasty material from the walls of the vessel; this is valuable when handling materials that burn in contact with a hot surface. The anchor paddle may also be used in a simple crystallising pan.

(*C*) *Double Motion Paddles.* For stirring very large batches of slow settling slurries in tanks of 40 ft or more in length, the paddle is mounted on a moving overhead crane. In the baker's mixer (Fig. 26.5) the paddle moves round the circular pan giving intense mixing of suspensions. In some cases, the vessel moves as well as the agitating device.

(*D*) *Kneaders.* For handling very thick pastes, two heavy Z-shaped blades are caused to rotate in opposite directions, as in Fig. 26.6. The clearance between the blades and the walls should be very small, particularly when the mix is being heated. On the other hand, with thick explosive pastes the clearance must be increased. This type of machine is usually tilted to facilitate emptying, and in all cases it is very heavily made, to stand up to the heavy stresses that arise during the handling of plastic magmas.

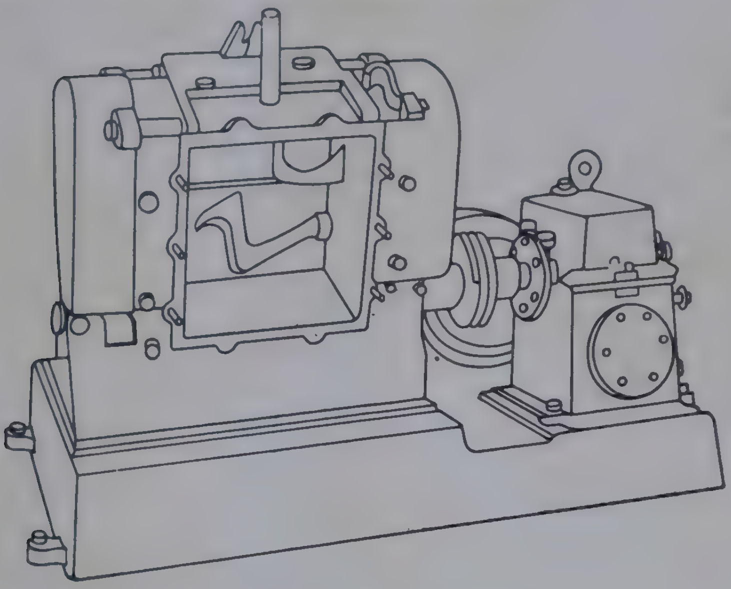


Fig. 26.6. Kneader

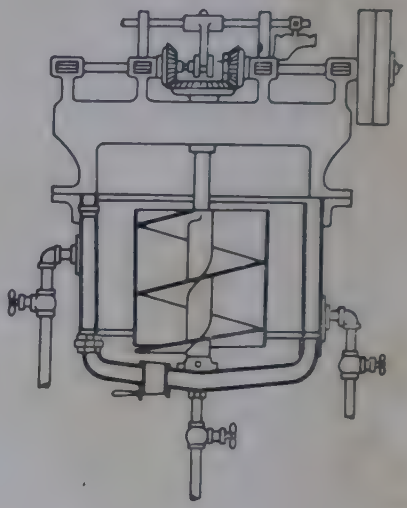


Fig. 26.7. Soap crutcher

(*E*) *Screw Type Mixers.* A paddle in the form of a vertical screw is used for suspending solids in liquids. The material is taken up by the screw and falls down by gravity. The soap crutcher, consisting of a vertical helix in a draft tube, is a modification of this type of mixer (Fig. 26.7). It is suitable for pastes like soap or paper pulp, and for some plastic mixtures.

(*F*) *Trough Mixers.* A simple horizontal trough fitted with a star paddle or ribbon element is useful for mixing solids, and may be used for incorporating solids into moderately thick pastes. It is particularly useful when heating is required as the trough can easily be jacketted (Fig. 26.8).

(*G*) *Special Forms.* Numerous special forms of equipment are used for particular operations. The Pug mill, used in the ceramic industry for mixing heavy clays, is shown in Fig. 26.9. The double cone mixer (Fig. 26.10) is used for the simple blending of granular powders, and the rolls described in Chapter 24 are used for intimate mixing of solids with liquids, as in ink manufacture, and the addition of fillers to rubber. For simple blending, powders may be

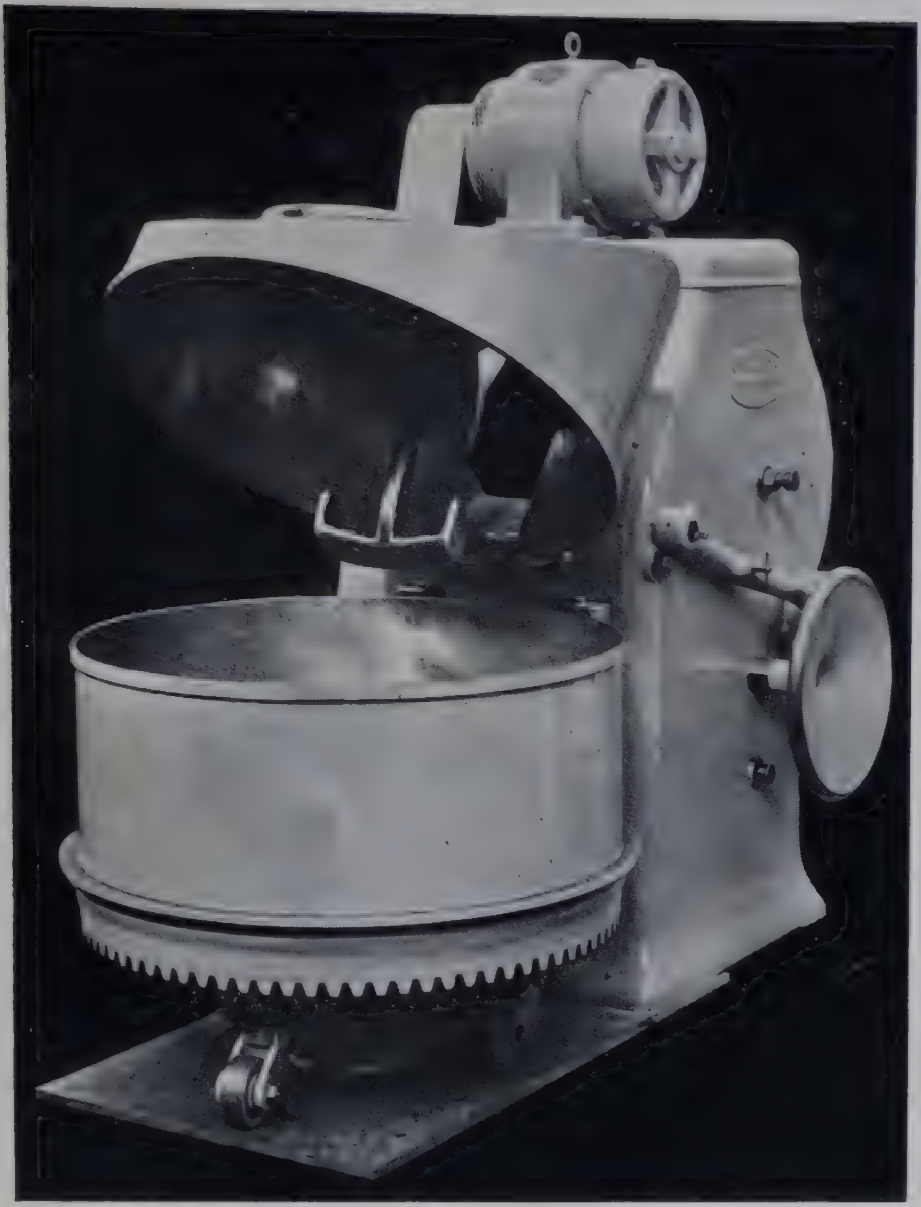


Fig. 26.5. Double-motion paddle mixer

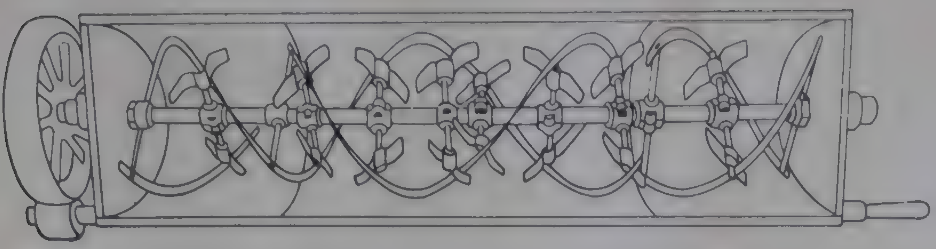


Fig. 26.8. Trough mixer

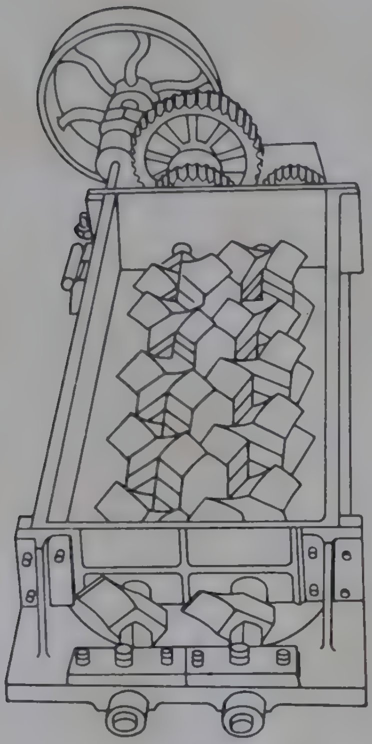


Fig. 26.9. Pug mill

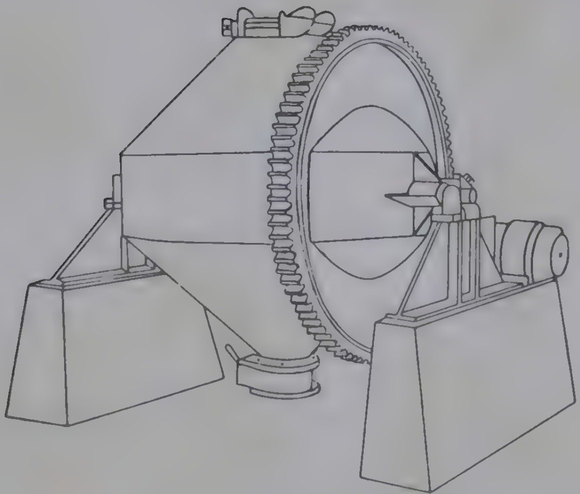
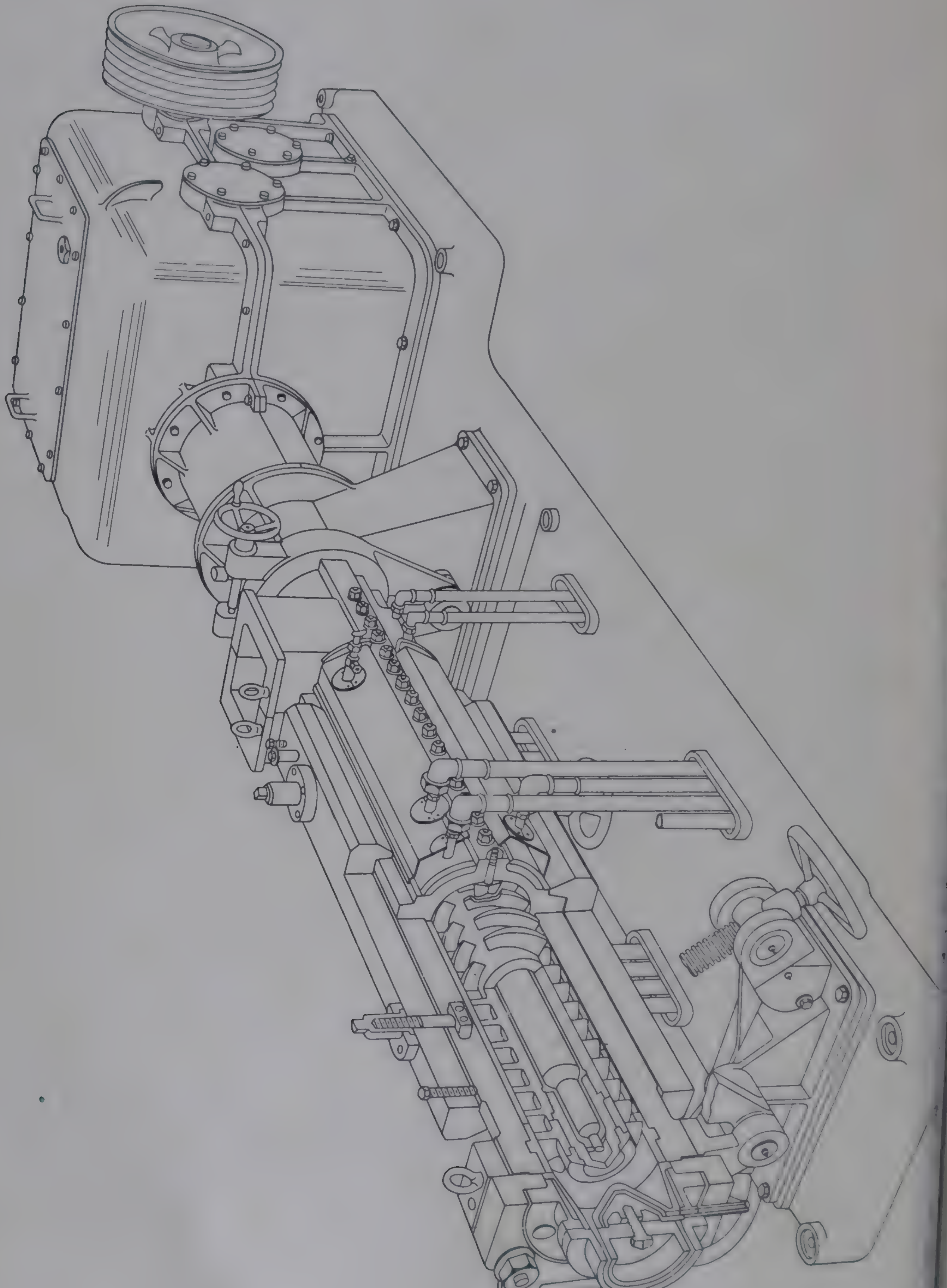


Fig. 26.10. Double cone mixer



allowed to fall down a tall tower fitted with a few baffles or screens. The Ko-Kneader (Fig. 26.11) is a new type of equipment which provides for continuous mixing of pastes and other materials. Here a worm runs in a horizontal casing fitted with teeth, and the worm is interrupted at intervals by gaps. The special feature of this machine is that an axial movement is given to the worm so that it can pass the teeth when the gap in the worm is in line with the teeth. In this way, material carried forward by the worm is given a kneading motion at each tooth, and intensive mixing is obtained.

(*H*) *Jet Mixers.* Mixing of gases, and sometimes liquids, can be effected by passing them together through a simple jet, or by the use of a jet mixer which is similar to an ejector (see Chapter 5). Mixing of liquids and the suspension of solids in liquids can also be carried out by blowing compressed air into the tank. Some details of this method of mixing have been given by FOSSETT(16).

## The Mixing of Liquids and Solids with Propellers or Turbines

In the above section, various types of mixing equipment have been described. For the great majority of reaction vessels, and for most operations involving liquid-liquid, and liquid-solid mixing, it is possible to use a propeller or turbine in a tank. This particular form of equipment will now be considered.

REAVELL(15) has suggested that this type of mixing can be divided into three classes:

(1) Liquids, with or without solids, which remain free flowing when mixing is complete; e.g. water and salt, acid and sand, light or medium oils.

(2) Liquids, with or without solids, which are viscous but still pourable when mixing is complete; e.g. heavy oils, paints, resins, syrups.

(3) Liquids, with solids, which form stiff pastes; e.g. core sands and binders, oil bound distempers, white lead and oil, putties.

The usual form of equipment is a vertical cylindrical tank, with height one and a half times to twice the diameter, fitted with an agitator. When the thickness of the mix corresponds to class (3) above, it is necessary for the agitator to conform to the shape of the vessel, so that the action corresponds with that of the kneading machines already described. With thin liquids, high speed propellers of diameter about one third that of the vessel are suitable, and for thicker mixtures the propeller diameter is increased and its speed reduced. Thus, high speed propellers are run at from 600 to 1500 r.p.m., and the kneaders and scraping agitators at speeds down to 120 or even 60 r.p.m.

***Propeller in Cylindrical Tank.*** If a propeller is mounted centrally, there is a tendency for the lighter fluid (usually air) to be drawn in to form a vortex and for the degree of agitation to be reduced. The flow pattern should be as indicated in Fig. 26.12, where the stream leaving the propeller is moving with a high velocity and initially in a straight line. The outer part of the stream, shown as *E*, turns back on itself and re-enters the feed to the propeller, whilst the inner streams, as at *A*, are of much greater length. A particle in any one stream will enter the next at the inlet side of the propeller and effective mixing occurs, a considerable up and down motion being provided. The agitation is strongest near the propeller and dead spaces form at the bottom of the tank. With this arrangement the unsupported length of the propeller shaft should not exceed 6 ft. If the contents of a very large vessel are to be stirred with a propeller of this kind, a foot bearing is essential. Despite a considerable amount of practical experimentation, these foot bearings usually give trouble, since corrosive liquids and solvents are frequently used so that it is very difficult to lubricate the bearings. It has been shown by REAVELL(15) that the fitting of a cruciform baffle at the bottom of the vessel (Fig. 26.13) enables much better

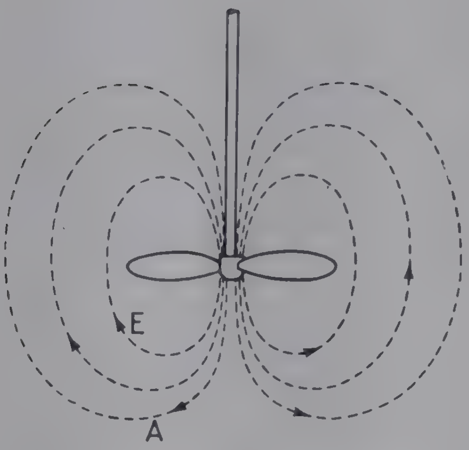


Fig. 26.12. Flow pattern from propeller mixer

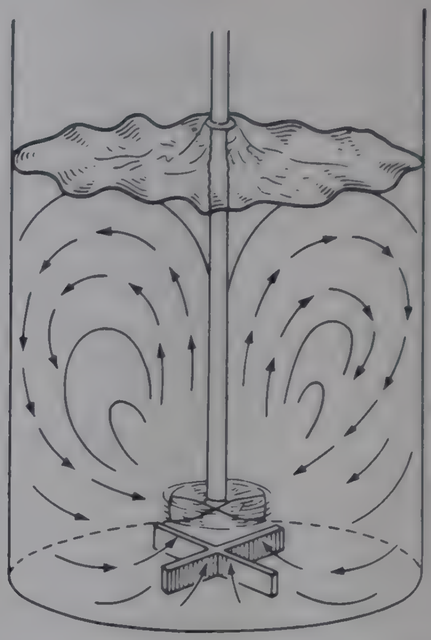


Fig. 26.13. Flow pattern in vessel with cruciform baffle

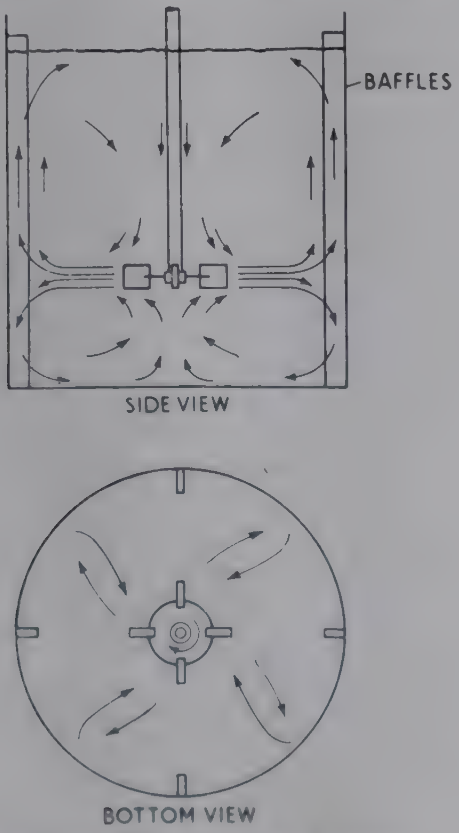

BAFFLES
SIDE VIEW
BOTTOM VIEW

Fig. 26.14. Flow pattern in vessel with vertical baffles

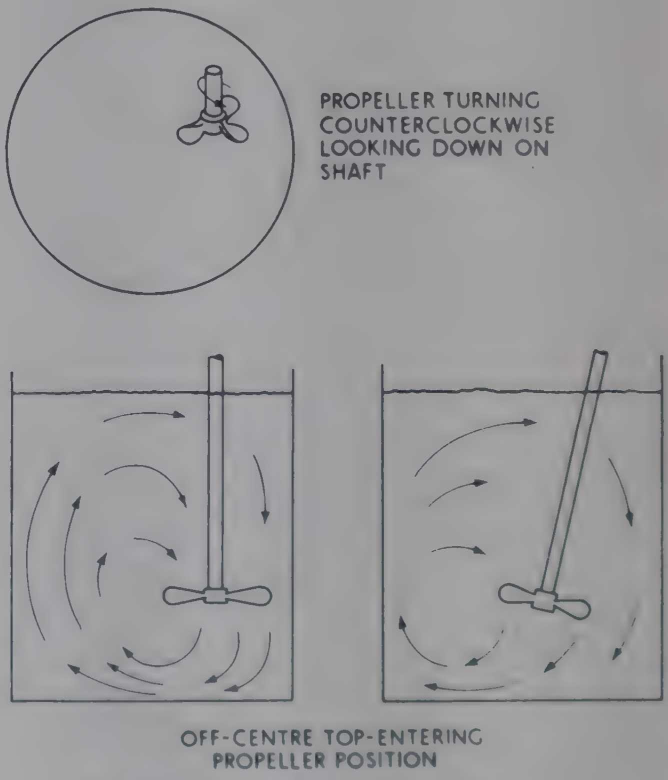

PROPELLER TURNING COUNTERCLOCKWISE LOOKING DOWN ON SHAFT
OFF-CENTRE TOP-ENTERING PROPELLER POSITION

Fig. 26.15. Flow pattern with agitator offset from centre

dispersion to be obtained. The rotor is arranged to force the fluid downwards; this arrangement gives much better axial flow and avoids the development of rotational movement of the liquid. The great reduction in the side-thrust on the shaft enables longer shafts of up to 10 ft to be used without foot-step bearings. To improve the rate of mixing and to minimise vortex formation, baffles are usually added. These take the form of thin vertical strips mounted against the walls of the vessel, as shown in Fig. 26.14. They considerably increase the power requirement, as discussed later. The off-setting of the agitator is another method of minimising vortex formation (Fig. 26.15).

***Portable Mixers.*** For a wide range of applications, a portable mixer which can be clamped on the top or side of the vessel is now used. This is commonly fitted with two propeller blades so that the bottom rotor forces the liquid upwards and the top rotor forces the liquid downwards. This form of unit can be supplied with about 3 horse power, but the size of the motor becomes too great at higher powers. To avoid excessive strain on the armature, some form of flexible coupling should be fitted between the motor and the main propeller shaft. Units of this kind are usually driven at a fairly high rate (800 r.p.m.), but a reduction gear can fairly easily be fitted to the unit for low speed operation.

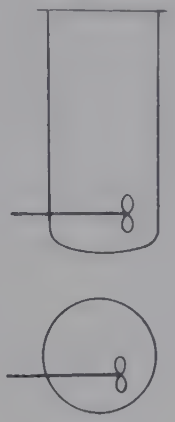


Fig. 26.16. Horizontally mounted propeller

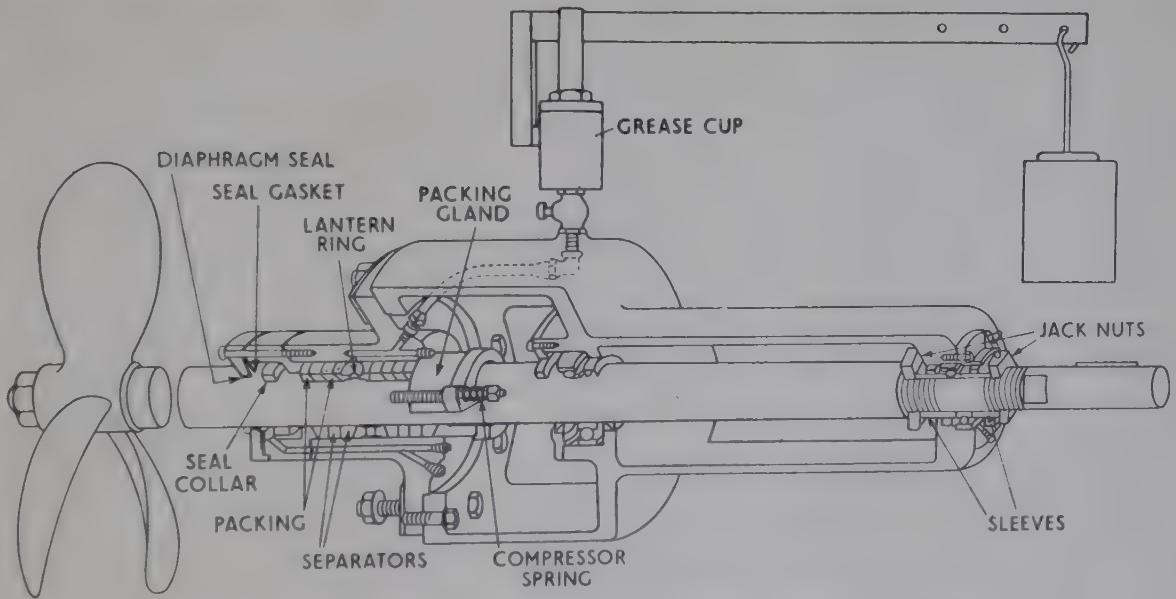


Fig. 26.17. Gland fittings for horizontally mounted propeller

***Horizontally Mounted Propellers.*** A propeller mounted on a horizontal shaft, positioned eccentrically as shown in Fig. 26.16, enables the contents of a very large tank to be stirred with a single propeller. This type of unit has been developed for very large power inputs (25 H.P.), but a good gland is needed where the shaft enters the vessel (Fig. 26.17).

***Turbine Rotors.*** The ordinary propeller may be replaced by a turbine which may be open or shrouded, the latter type being much more expensive. The flow pattern with a turbine, as shown in Fig. 26.18, is quite different from that obtained with a marine propeller. Turbines can be used for rather more viscous materials than propellers, but the power consumption is much greater. In comparing a propeller and a turbine we may note:

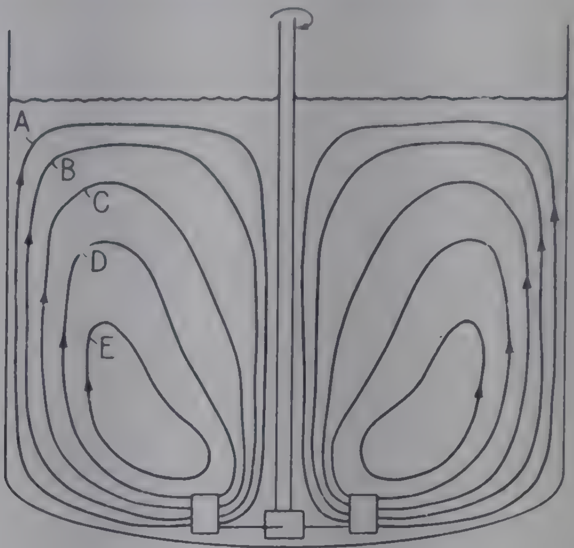


Fig. 26.18. Flow pattern with turbine rotor

*Propellers:*

(1) are self cleaning in operation,
(2) can be used at a wide range of speeds,
(3) give excellent shearing effect at high speeds,
(4) do not damage dispersed particles at low speeds,
(5) are reasonably economical in power, provided the pitch is adjusted according to the speed,
(6) by offset mounting, avoid vortex formation,
(7) if horizontally mounted, require a stuffing box in the liquid, and
(8) are not effective in viscous liquids.

*Shrouded Turbines:*

(1) are excellent for providing circulation,
(2) are normally mounted on a vertical shaft with the stuffing box above the liquid,
(3) are effective in fluids of high viscosity,
(4) are easily fouled or plugged by solid particles,
(5) are expensive to fabricate,
(6) are restricted to a narrow range of speeds, and
(7) do not damage dispersed particles at economical speeds.

*Open Impellers:*

(1) are less easily plugged than the shrouded type,
(2) are less expensive, and
(3) give a less well controlled flow pattern.

***Propellers with Coil.*** If a coil is used in the tank to provide cooling, then the geometrical arrangements commonly adopted are as in Fig. 26.19. This set-up is very common for reaction vessels in the organic chemical industry.

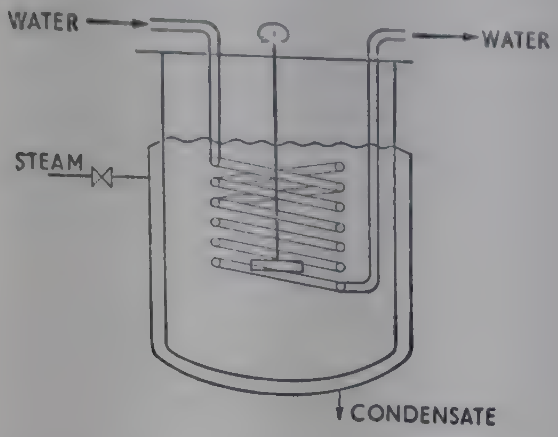


Fig. 26.19. Reaction vessel with jacket and coil

## Rate of Mixing and Power Consumption of Agitators

In considering the speed of rotation of an agitator, a compromise is usually made between a high speed which gives rapid mixing and a lower speed where a smaller power is required. One of the earliest publications in which these quantities were discussed is by Wood, Whittemore and Badger[(3)] who used a

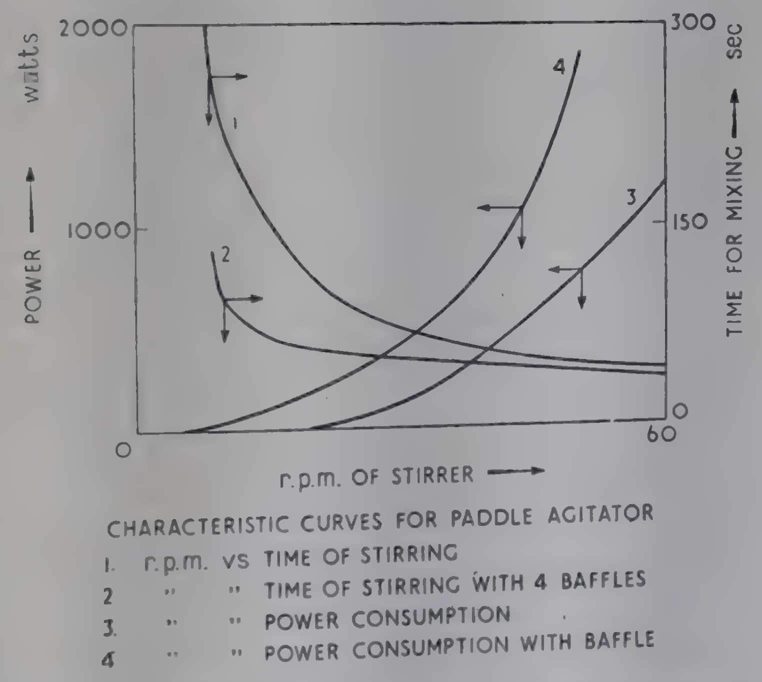


Fig. 26.20. Power and time of mixing as a function of speed for paddle agitator

600 gallon vertical tank of 5 ft diameter, fitted with a simple paddle of 4 in. × 4 in. section. Strong brine was first run into the tank, and water was then run in on top. The power for mixing was measured electrically, but accurate results were difficult to obtain because of the difficulty of assessing the power used in the belt drive and gears. Some of their results are shown in Fig. 26.20. From curve 1, it is seen that the time of stirring fell off quite steadily with an increase in speed. The addition of four simple baffles (1 in. × 4 in. positioned 2 in. from

the wall), reduced the time for stirring but increased the power requirement (curve 2). The degree of mixing was assessed by taking samples from various points and measuring their electrical conductivity. Curves 3 and 4 show the change in power consumption at various speeds with and without baffles.

HIXSON and WILKENS(5) measured the power absorbed by a four-bladed propeller with a 45° pitch, operating in a cylindrical vessel. The power consumption was expressed in terms of watts per 100 gallons plotted against speed. In general, they found that the power consumption per unit volume increased rapidly as the size of the system was increased.

STOOPS and LOVELL(7) examined the power consumption of a propeller agitator under various conditions. Three feasible methods for the determination of the power required were suggested:

1. Fitting some form of Prony brake to the shaft between the motor and the propeller. In this way, the mechanical power output can be measured for various conditions of operation.

2. Placing the mixing vessel on a turn-table and determining the torque necessary to prevent its rotation.

3. Connecting the driving shaft to the propeller by a spring or dynamometer system, and measuring the torque by the relative displacement of the two shafts.

The first method is the simplest, but they found that reliable results were very difficult to obtain as the mechanical energy was always a small proportion of the electrical energy. The second method has been used by WHITE and BRENNER(6), but is rather difficult to apply to large installations. The third method, although more complicated, has been found by many workers to give the most satisfactory results. Various forms of dynamometers have been used, and these are described by STOOPS and LOVELL(7) and BLACK(11).

***Fluid Motion and Power Requirements.*** One of the problems confronting the designer of agitating equipment is that of deducing from experimental work with small units what will be the most satisfactory arrangement for a larger unit. In order to achieve the same kind of flow pattern in two units, we must maintain geometrical, kinematic, and dynamic similarity, as well as maintain similar boundary conditions. This problem has been discussed by a number of workers, including RUSHTON *et al.*(13) and KRAMERS *et al.*(20). The latter paper represents virtually the only attempt to assess the relative merits of different arrangements of mixers. It has been found convenient to relate the power used by the agitator to the geometrical and mechanical arrangements of the mixer, and thus to obtain a direct indication of the change in power for any alteration to the mixer. The general method of attack is indicated in the following section, the nomenclature following that given in the diagram (Fig. 26.21).

For similarity in two mixing systems, we must arrange:—

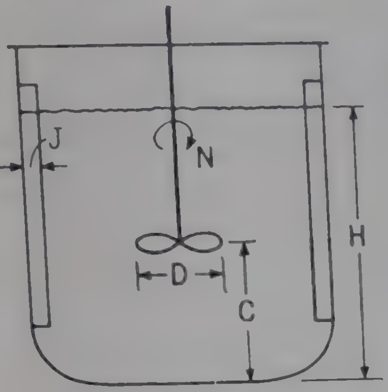
1. *Geometrical Similarity.* This will define the boundary conditions; corresponding dimensions will have the same ratio.

Thus we must have:—

$$\frac{D_T}{D};\quad \frac{C}{D};\quad \frac{J}{D};\quad \frac{S}{D};\quad \frac{W}{D};\quad \frac{H}{D};\quad \text{the same in the two systems.}$$

2. *Kinematic Similarity.* This requires that velocities at corresponding points must have the same ratio as those at other corresponding points. The paths of motion must also be alike.

3. *Dynamic Similarity.* This requires that the ratio of forces at corresponding points is equal to that at other corresponding points.



D DIAMETER OF AGITATOR
$D_T$ " " TANK
C HEIGHT OF AGITATOR FROM BASE OF TANK
H DEPTH OF LIQUID
R Nº OF BAFFLES
J WIDTH OF BAFFLES
N SPEED OF AGITATOR
S PITCH OF AGITATOR
W WIDTH OF BLADES OF AGITATOR OR PADDLE

Fig. 26.21. Dimensions of agitator

If the boundary conditions are fixed, then we can express one variable such as power $P$ in terms of a number of other independent variables

$$P = \phi(D,\mu,g,\rho,N) \qquad \ldots\ldots(26.1)$$

If we can write this as the product of powers of the variables,

$$P = K(D^a\,.\,\mu^b\,.\,g^e\,.\,\rho^f\,.\,N^j) \qquad \ldots\ldots(26.2)$$

Expressing these terms on the basis of the **L.M.T.** system of units,

$$\frac{\mathbf{M.L.}^2}{\mathbf{T}^3} = \left[\mathbf{L}^a\,.\left(\frac{\mathbf{M}}{\mathbf{L.T.}}\right)^b.\left(\frac{\mathbf{L}}{\mathbf{T}^2}\right)^e.\left(\frac{\mathbf{M}}{\mathbf{L}^3}\right)^f.\left(\frac{1}{\mathbf{T}}\right)^j\right]$$

If we now equate the dimensions on each side,

for **L** $\quad 2 = a - b + e - 3j$

**M** $\quad 1 = b + f$

**T** $\quad -3 = -b - 2e - j$

Writing these indices in terms of $b$ and $e$,

$$f = 1 - b$$

$$a = 2 + b - e + 3 - 3b = 5 - 2b - e$$

$$j = -b - 2e + 3$$

$$\therefore \quad P = K[D^{-2b-e+5}\,.\,\mu^b\,.\,g^e\,.\,\rho^{1-b}\,.\,N^{-b-2e+3}]$$

$$= K\left[\left(D^5\,.\,\rho\,.\,N^3\right).\left(\frac{D^2\,.\,N\,.\,\rho}{\mu}\right)^{-b}.\left(\frac{D\,.\,N^2}{g}\right)^{-e}\right]$$

$$\therefore \quad \frac{P}{D^5N^3\rho} = K\left[\frac{D^2\,.\,N\,.\,\rho}{\mu}\right]^{-b}.\left[\frac{DN^2}{g}\right]^{-e} \qquad \ldots\ldots(26.3)$$

or

$$\mathbf{N}_P = K\,.\,Re.^m\,.\,Fr.^r \qquad \ldots\ldots(26.3a)$$

In this analysis the Reynolds group $\dfrac{D^2 . N . \rho}{\mu}$ accounts for the viscous forces, and may be regarded as the ratio of the inertia to the viscous forces, and the Froude Group $\dfrac{D . N^2}{g}$ represents the influence of gravitation. Where, as is generally the case, the viscous forces are significant, then kinematic similarity will be obtained by arranging for *Re.* to be the same in the two systems. RUSHTON(13) has given data for a propeller (Fig. 26.22), by plotting the Power Number $\mathbf{N}_P$ vs *Re.*

For values of *Re.* $< 300$, all the data fall on a single line indicating that the Froude group has no important effect.

Thus $\quad \mathbf{N}_P = K . (Re.)^m$

and $\quad m = -1,$

so that $\quad P \doteq K . \mu . N^2 . D^3 \qquad \ldots .(26.4)$

where $K$ depends on the impeller and surroundings. For marine-type three-bladed propellers with pitch equal to diameter, $K$ is found to have a value of about 41.

For higher values of *Re.*, the Froude group plays a part, and separate lines are drawn for various speeds. The Reynolds group was varied by using different viscosities as well as different speeds, and the slanting lines represent conditions of constant viscosity.

Fig. 26.23, also taken from the work of RUSHTON, shows similar data for a 6 in. diameter turbine with six flat blades. The effect of introducing baffles is also brought out in this chart.

BISSELL *et al.*(10) have given the following data for the power consumption with different baffles for a turbine mixer situated one diameter from the bottom of a cylindrical container, and operating at a Reynolds number *Re.* of $5 \times 10^4$.

TABLE 26.1. *Effect of width and number of baffles on power*

| Baffle width as % of tank diameter | % power based on 4 baffles of $\frac{1}{12}$ tank diameter | | | | | | |
|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | baffles |
| 2 | 30 | 52 | 63 | 72 | 76 | 78 | |
| 5·5($\frac{1}{18}$) | 40 | 64 | 78 | 87 | 92 | 94 | |
| 8·3($\frac{1}{12}$) | 50 | 78 | 92 | 100 | 102 | 104 | |
| 10·0($\frac{1}{10}$) | 58 | 82 | 95 | 103 | 105 | 106 | |

The power requirement of a turbine mixer operating at a peripheral speed of 700 ft/min for different diameters is given by the figures below in Table 26.2(1). The effects of peripheral speed and viscosity of the liquid are also given.

TABLE 26.2*a*. *Effect of impeller diameter on power for turbine mixer*

| Diameter in inches | Horse power |
|---|---|
| 6 | 0·1 |
| 10 | 0·3 |
| 20 | 1·1 |
| 40 | 4 |
| 80 | 15 |

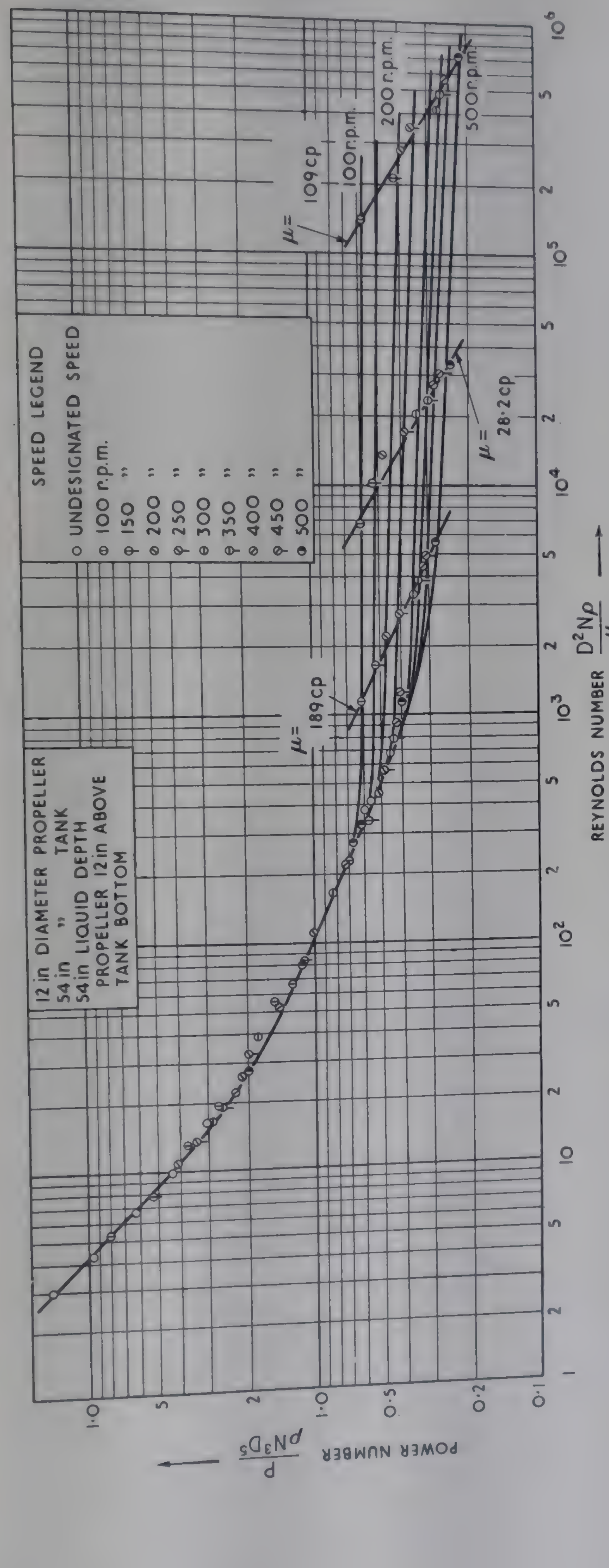


Fig. 26.22. Power number as a function of Reynolds number for a propeller mixer

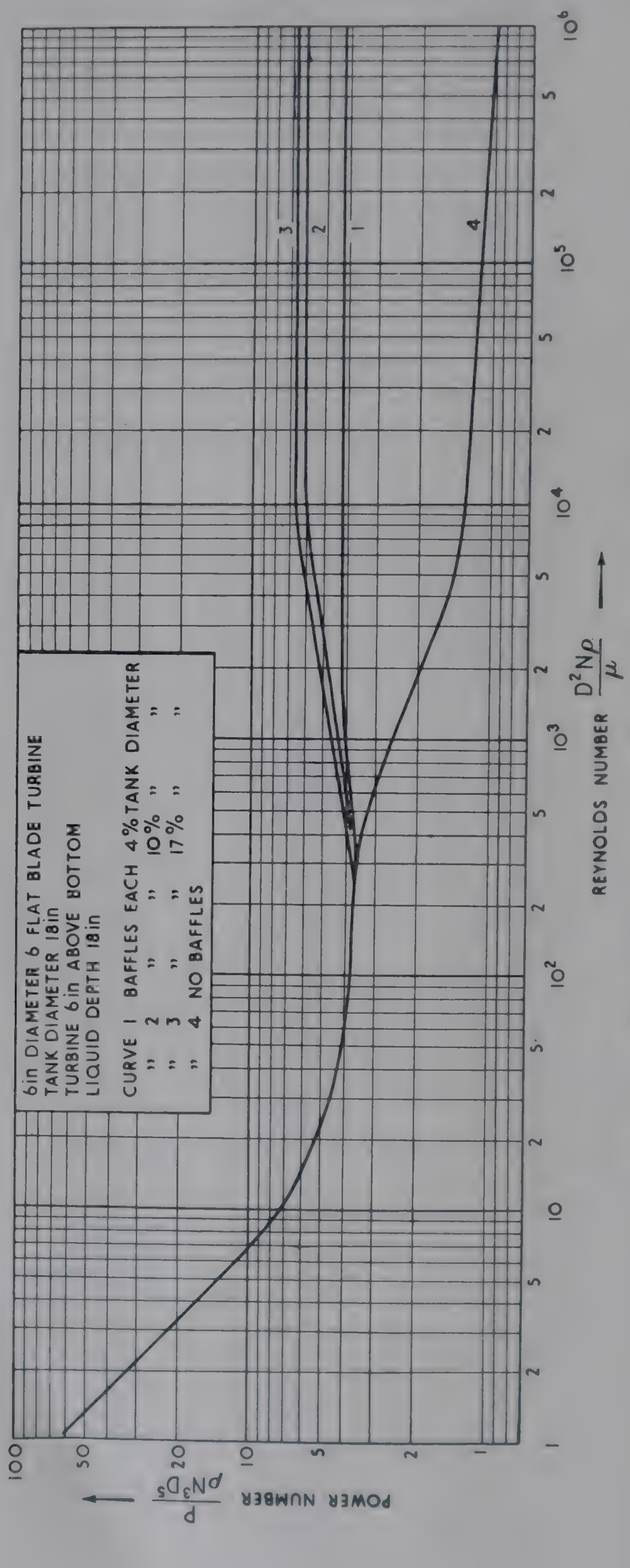


Fig. 26.23. Power number as a function of Reynolds number for a turbine mixer

TABLE 26.2*b*. *Effect of liquid viscosity on power for turbine mixer*

| $\mu$ in centipoises | Power (as percentage of that for 1 cp) |
|---|---|
| 1 | 100 |
| 1,000 | 120 |
| 10,000 | 130 |
| 50,000 | 210 |

TABLE 26.2*c*. *Effect of peripheral speed on power for turbine mixer*

| Peripheral speed ft/min | Power (as percentage of that for 700 ft/min). |
|---|---|
| 130 | 1 |
| 320 | 10 |
| 550 | 50 |
| 700 | 100 |
| 900 | 200 |
| 1,200 | 520 |

***The Rate of Mixing.*** In assessing the performance of a given unit, it is not only the power used that is important, but also the rate at which the desired degree of mixing is achieved. It has proved very difficult to define a scale for expressing the degree of mixing achieved at any time, and the final decision as to when a material is mixed is still left to the skill, experience and judgement of the operator. The essential test is the practical one of whether the mixture meets the requirement for which it is being prepared. This may be measured, for example, by the rate of burning of a blended nitrocellulose propellant, or by the colour reflection of a paint, or by the uniformity of conduction of an electrolyte in water. These are varying scales which cannot serve as a general criterion of performance. A few of the methods tried for assessing the effectiveness of agitation are:

(1) The rate of dispersion of an electrolyte, such as brine, in water.

(2) The rate of distribution of sand in water.

(3) The rate of dissolution of solids in different solvents.

Method (1) was tried by WOOD, WHITTEMORE and BADGER(3) and more recently by KRAMERS *et al.*(20). KRAMERS used two tanks of 0·32 and 0·64 metres diameter and a three-bladed marine propeller of diameter one quarter that of the tank. To a solution of potassium chloride containing about 3 mg/cm$^3$, a small quantity of concentrated solution containing 100 mg/cm$^3$ was added. By using a suitable electronic arrangement, a record was obtained of the change in concentration with time; the time to reach an arbitrary standard of 0·1% deviation was taken as the mixing time. Typical records are illustrated in Fig. 26.24.

Method (2) has not been widely used but, as suggested by REAVELL[15], if polyvinyl chloride particles are used, it is possible to observe the flow pattern of an agitator. In method (3), suggested by HIXSON and CROWELL[4], when the change in concentration is small then the rate of solution is

$$\frac{dw}{dt} = -kw^{2/3} \qquad \ldots.(26.5)$$

where $w$ is the weight of solid solute present at time $t$.

i.e. $$k't = w_0^{1/3} - w^{1/3}. \qquad \ldots.(26.6)$$

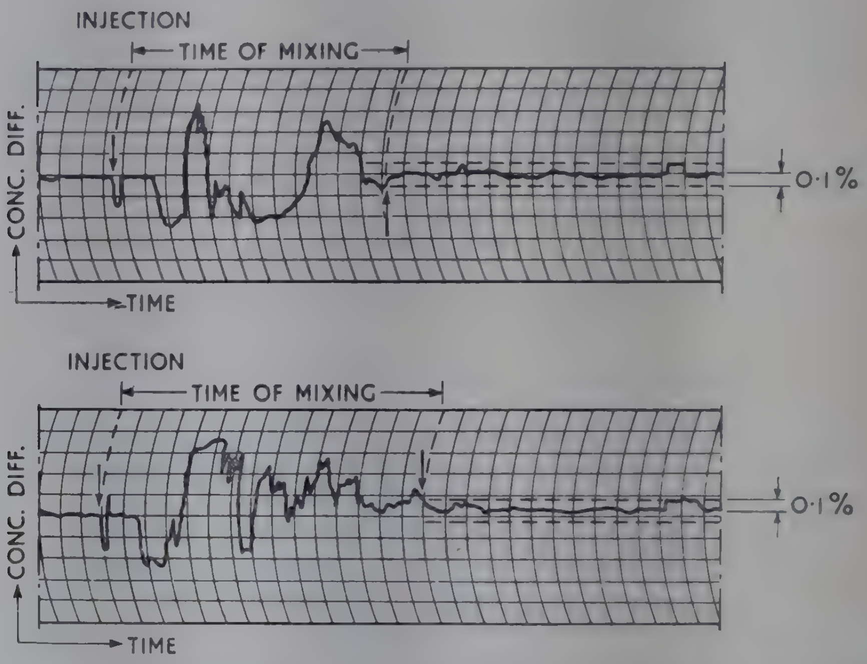


Fig. 26.24. Determination of time of mixing by method of Kramers

## Mixing of Solid Particles

This particular problem has been examined by two or three workers who have simplified matters by using only two materials and simple forms of mixers. Their work, which is discussed below, brings out some of the difficulties in understanding the process of mixing, and in formulating a useful scale for expressing performance.

In the mixing of solid particles, three mechanisms may be involved:

(1) convective mixing, in which groups of particles are moved from one position to another,

(2) diffusion mixing, where the particles are distributed over a freshly developed interface, and

(3) shear mixing, where slipping planes are formed.

These three mechanisms will occur to varying extents in different kinds of mixers and with different kinds of particles. A trough mixer with a ribbon spiral (Fig. 26.8) will give almost pure convective mixing, but a simple barrel-mixer (Fig. 26.27) will give mainly a form of diffusion mixing.

***The Degree of Mixing.*** It is difficult to express the degree of mixing, but any index should be related to the properties of the required mix, should be easy to measure, and should be suitable for a variety of different mixers. When dealing with solid particles, the statistical variation in composition among samples withdrawn at any time from a mix is commonly used as a measure of the degree of mixing. The standard deviation $s$, (i.e. the square root of the mean of the squares of the individual deviations) or the variance $s^2$ is generally used. A particulate material cannot attain the perfect mixing that is possible with two fluids, for the best that can be obtained will be a degree of randomness in which two similar particles may well be side by side. No amount of mixing will lead to the formation of a uniform mosaic (Fig. 26.25), but only to a condition, such as Fig. 26.26, where there is an overall uniformity but not point uniformity. For a completely random mix of uniform particles distinguishable, say, only by colour, LACEY[8, 21] has shown that

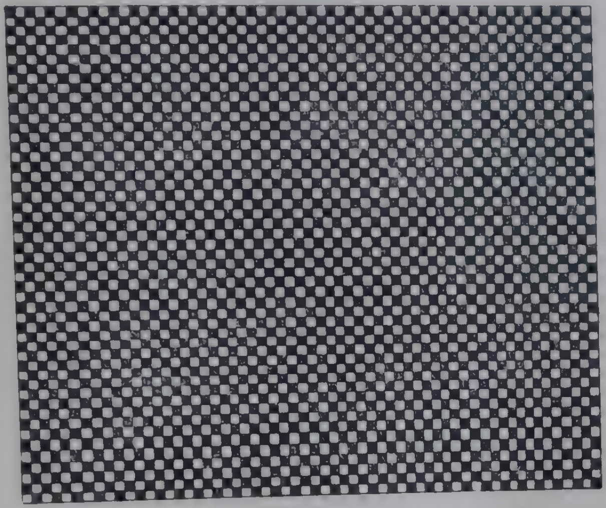


Fig. 26.25. Uniform mosaic

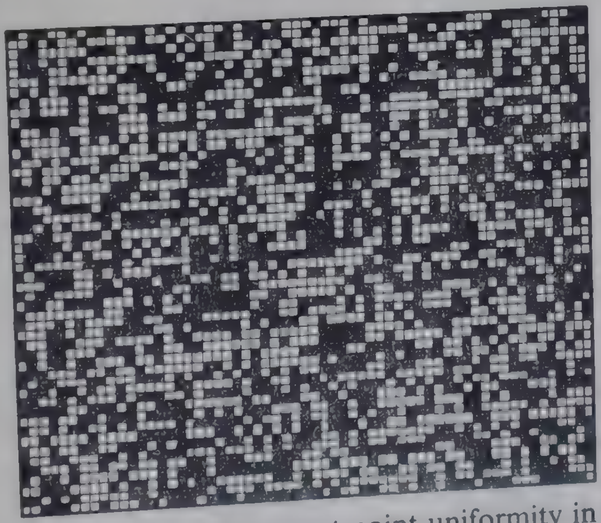


Fig. 26.26. Overall but not point uniformity in mix

$$s_r^2 = \frac{p(1-p)}{n} \qquad \ldots.(26.7)$$

where $s_r^2$ is the variance for the mixture, $p$ is the overall proportion of particles of one colour, and $n$ is the number of particles in each sample.

This at once brings out the importance of the size of the sample in relation to the size of the particles. In an incompletely randomized material, $s^2$ will be greater, and in a completely unmixed system (indicated by suffix $o$) it can be shown that

$$s_o^2 = p(1-p) \qquad \ldots.(26.8)$$

which is independent of the number of particles. Only a definite number of samples can in practice be taken from a mixture, and hence $s$ will itself be subject to random errors. The above analysis has been extended to systems containing particles of different sizes by BUSLIK(14).

When a material is partly mixed, then the degree of mixing can be represented by some term $M$, and several methods have been suggested for expressing $M$ in terms of measurable quantities. If $s$ is obtained from examination of a large number of samples then, as suggested by LACEY, we can define $M$ as equal to $s_r/s$ or, as more recently suggested by KRAMERS(22), $M = \dfrac{s_o - s}{s_o - s_r}$, where, as before, $s_o$ is the value of $s$ for the unmixed material. This form of expression is useful in that $M = 0$ for an unmixed material and 1 for a completely randomized material ($s = s_r$). If we use $s^2$ instead of $s$, then we can rearrange the above expression to give:—

$$M = \frac{s_o^2 - s^2}{s_o^2 - s_r^2}$$

or

$$1 - M = \frac{s^2 - s_r^2}{s_o^2 - s_r^2} \qquad \ldots.(26.9)$$

For diffusive mixing, $M$ will be independent of sample size provided the sample is small. For convective mixing, KRAMERS has suggested that we have groups of particles randomly distributed, each group behaving as a unit containing $n_g$ particles. As mixing proceeds $n_g$ becomes smaller. The number of groups will then be $n/n_g$, where $n$ is the number of particles in each sample. Applying equation 26.7, we get

$$s^2 = \frac{p(1-p)}{n/n_g} = n_g s_r^2$$

and this gives

$$1 - M = \frac{n_g s_r^2 - s_r^2}{n s_r^2 - s_r^2} = \frac{n_g - 1}{n - 1} \qquad \ldots.(26.10)$$

Thus, with convective mixing, $1 - M$ will depend on the sample size.

***The Rate of Mixing.*** Expressions for the rate of mixing can be developed for any one of the possible mechanisms. Since mixing involves obtaining an equilibrium condition of uniform randomness, we may expect the relation between $M$ and time to be of the general form,

$$M = 1 - e^{-ct} \qquad \ldots.(26.11)$$

where $c$ is some constant depending on the nature of the particles and the physical action of the mixer.

Imagine a cylindrical vessel (Fig. 26.27) in which one substance $A$ is poured into the bottom and the second $B$ on top. Then the boundary surface between the two materials is a minimum. The process of mixing consists in making some of $A$ enter the space occupied by $B$, and some of $B$ enter the lower section originally filled by $A$. We may consider this as the diffusion of $A$ across the initial boundary into $B$, and of $B$ into $A$. We can imagine this process to continue till there is a maximum degree of dispersion, and a maximum interfacial area between the two materials. This type of process is somewhat akin to that of diffusion, and we may tentatively apply the relationship given by Fick's Law (equation 7.1 or 18.77 or 19.2) in the following way.

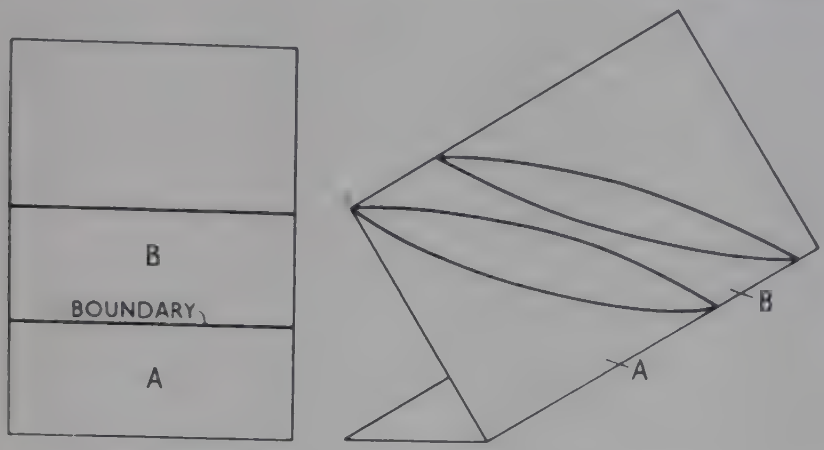


Fig. 26.27. Initial arrangement in mixing test

Let $A$ be the area of the interface per unit volume of the mix, and $A_m$ be the maximum interfacial surface per unit volume that can be obtained.

Then
$$\frac{\mathrm{d}A}{\mathrm{d}t} = c(A_m - A) \qquad \ldots.(26.12a)$$

and
$$A = A_m(1 - e^{-ct}) \qquad \ldots.(26.12b)$$

Suppose that, after any time $t$, a number of samples are removed from the mix, and are examined to see how many contain both components. If a sample contains both components, it will contain an element of the interfacial surface. If $Y$ is the fraction of the samples containing the two materials in approximately the proportion in the whole mix, we can then write

$$Y = 1 - e^{-ct} \qquad \ldots.(26.13)$$

COULSON and MAITRA(12) have examined the mixing of a number of pairs of materials in a simple drum mixer, and have expressed their results in the form of a plot of $\ln \frac{100}{X}$ vs $t$, where $X$ is the percentage of the samples that are unmixed, i.e. $Y = 1 - \frac{X}{100}$. They were able to show that the constant $c$ depends on:

(1) the total volume of the material,
(2) the inclination of the drum,
(3) the speed of rotation of the drum,
(4) the particle size of each component,
(5) the density of each component, and
(6) the relative volume of each component.

Whilst the precise values of *c* are only of value for the particular mixer under examination, they do bring out the effect of these variables. Thus, Fig. 26.28 shows the effect of speed of rotation, and the best results are obtained when the mixture is just not taken round by centrifugal action. If fine particles are put in at the bottom and coarse on the top, then on rotation no mixing occurs, the coarse remaining on top. If the coarse particles are put at the bottom and the fine on top, then on rotation mixing occurs to an appreciable extent, but on further rotation *Y* falls, and the coarse particles settle out on the top. This is shown in Fig. 26.29 which shows a maximum degree of mixing. Again, with particles of the same size but differing density, the denser migrate to the

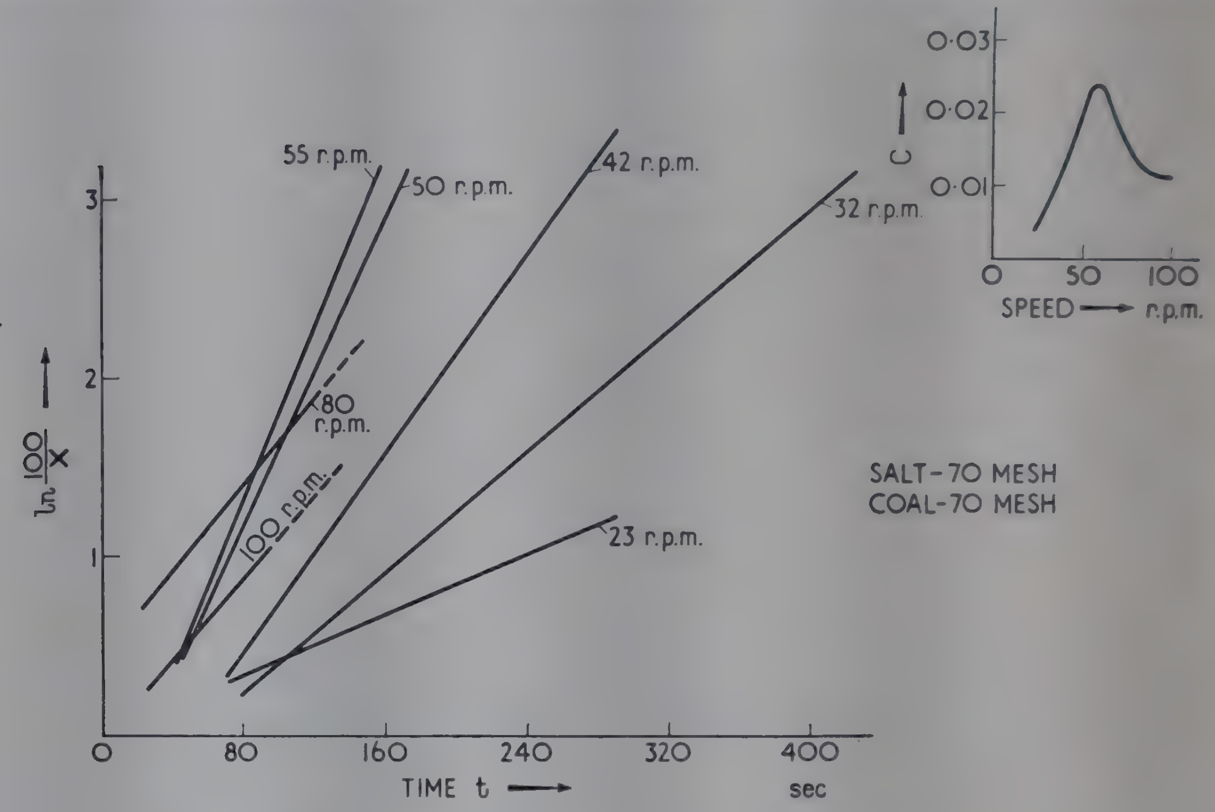


Fig. 26.28. Effect of speed of rotation on rate of mixing

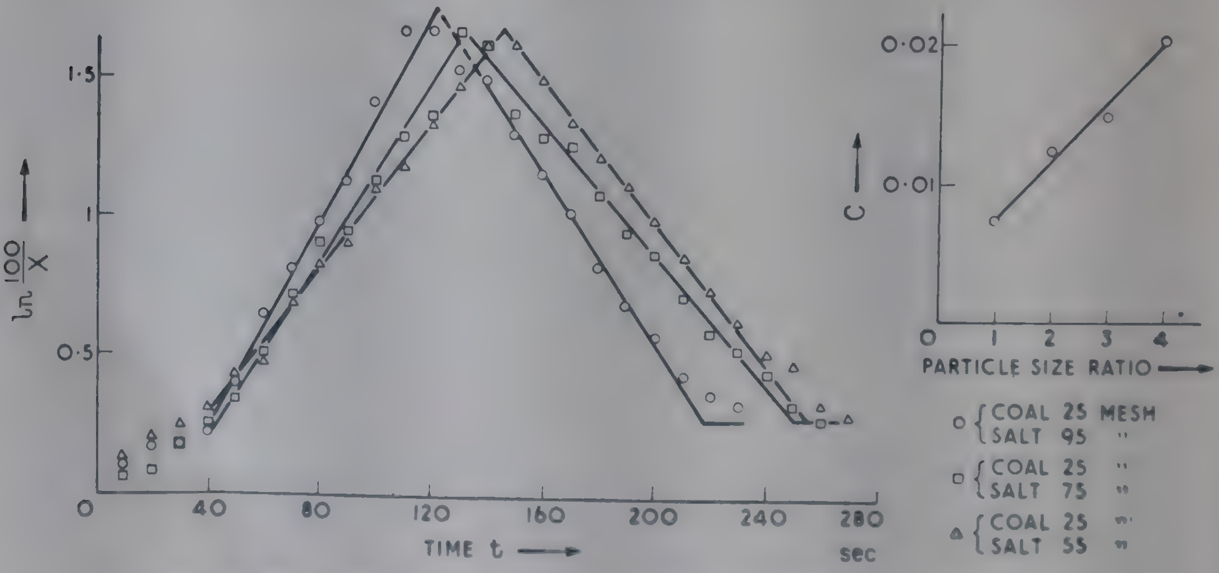


Fig. 26.29. Mixing and subsequent separation of solid particles

bottom and the lighter to the top. Thus, if the lighter particles are put in at the bottom and the heavier at the top, rotation of the drum will give improved mixing to a definite value, after which the heavier particles will settle to the bottom. This kind of analysis of simple mixers has been made by Blumberg and Maritz(19) and by Kramers(22), but the results of their experiments are little more than qualitative.

Brothman *et al.*(9) have given an alternative theory for the process of mixing of solid particles, which has been described as a shuffling process. In their view, as mixing takes place there will be an increasing chance that a sample of a given size intercepts more than one part of the interface, and hence the number of mixed samples would increase less rapidly than the surface. This is further described by Herdan(2). The application of some of these ideas to continuous mixing has been attempted by Danckwerts(17, 18).

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## LIST OF SYMBOLS FOR CHAPTER 26

| | | |
|---|---|---|
| $A$ | Interfacial area between constituents | $\mathbf{L}^2$ |
| $A_m$ | Minimum interfacial area | $\mathbf{L}^2$ |
| $a$ | Index | — |
| $b$ | Index | — |
| $C$ | Height of agitator from bottom of tank | $\mathbf{L}$ |
| $c$ | Coefficient of $t$ | $\mathbf{T}^{-1}$ |
| $D$ | Diameter of agitator | $\mathbf{L}$ |
| $D_T$ | Diameter of tank | $\mathbf{L}$ |
| $e$ | Index | — |
| $f$ | Index | — |
| $g$ | Acceleration due to gravity | $\mathbf{LT}^{-2}$ |
| $H$ | Depth of liquid | $\mathbf{L}$ |
| $J$ | Width of baffles | $\mathbf{L}$ |
| $j$ | Index | — |
| $K$ | Constant | — |
| $k, k'$ | Coefficients | $\mathbf{M}^{1/3}\mathbf{L}^{1/3}\mathbf{T}^{-5/3}$ |
| $M$ | Degree of mixing | — |
| $m$ | Index | — |
| $N$ | Revolutions per unit time | $\mathbf{T}^{-1}$ |
| $\mathrm{N}_P$ | Power number | — |
| $n$ | Number of particles in each sample | — |
| $n_g$ | Number of particles in a group | — |
| $P$ | Power | $\mathbf{ML}^2\mathbf{T}^{-3}$ |
| $p$ | Overall proportion of particles of one colour | — |
| $R$ | Number of baffles | — |
| $r$ | Index | — |
| $S$ | Pitch of agitator | $\mathbf{L}$ |
| $s$ | Standard deviation | — |
| $s_r$ | Standard deviation of random mixture | — |
| $s_o$ | Standard deviation of unmixed material | — |
| $t$ | Time | $\mathbf{T}$ |
| $W$ | Width of blades of agitator or paddle | $\mathbf{L}$ |
| $w$ | Weight of solute at time $t$ | $\mathbf{MLT}^{-2}$ |
| $w_0$ | Weight of solute at time $t = 0$ | $\mathbf{MLT}^{-2}$ |
| $X$ | Percentage of samples in which particles are not mixed | — |
| $Y$ | Fraction of samples in which particles are mixed | — |
| $\mu$ | Viscosity | $\mathbf{ML}^{-1}\mathbf{T}^{-1}$ |
| $\rho$ | Density | $\mathbf{ML}^{-3}$ |
| *Fr.* | Froude number | — |
| *Re.* | Reynolds number | — |

# Appendix

# Problems

(A number of these questions are taken from examination papers of the University of London)

1. A slurry, containing 0·2 lb of solid (specific gravity 3·0) per pound of water, is fed to a rotary drum filter, 2 ft long and 2 ft in diameter. The drum rotates at one revolution in six minutes and 20% of the filtering surface is in contact with the slurry at any given instant. If filtrate is produced at the rate of 1000 lb/hr and the cake has a voidage of 0·5, what thickness of cake is formed when filtering with a vacuum of 20 in. of mercury?

The rotary filter breaks down and the operation has to be carried out temporarily in a plate and frame press with frames 1 ft square. The press takes 2 minutes to dismantle and 2 minutes to re-assemble, and, in addition, 2 minutes are required to remove the cake from each frame. If filtration is to be carried out at the same overall rate as before, with an operating pressure of 25 lb/in.$^2$-G., what is the minimum number of frames that must be used and what is the thickness of each? Assume the cakes to be incompressible and neglect the resistance of the filter media.

2. A slurry containing 100 gm of whiting, of specific gravity 3·0, per litre of water, is filtered in a plate and frame press, which takes 15 minutes to dismantle, clean and re-assemble. If the filter cake is incompressible and has a voidage of 0·4, what is the optimum thickness of cake for a filtration pressure of 150 lb/in.$^2$? If the cake is washed at 75 lb/in.$^2$ and the total volume of wash water employed is $\frac{1}{4}$ of that of the filtrate, how is the optimum thickness of cake affected? Neglect the resistance of the filter medium and take the viscosity of water as 1 centipoise. In an experiment, a pressure of 24 lb/in.$^2$ produced a flow of water of 1 cc per minute through a centimetre cube of filter cake.

3. A plate and frame press, filtering a slurry, gave a total of 283 ft of filtrate in 30 minutes and 400 ft$^3$ in 60 minutes, when filtration was stopped. Estimate the washing time in minutes if 100 ft$^3$ of wash water are used. The resistance of the cloth can be neglected and a constant pressure is used throughout.

4. In the filtration of a certain sludge the initial period is effected at a constant rate with the feed pump at full capacity, till the pressure reaches 60 lb/in.$^2$ The pressure is then maintained at this value for the remainder of the filtration. The constant rate operation requires 15 minutes and one third of the total filtrate is obtained during this period.

Neglecting the resistance of the filter medium, determine (*a*) the total filtration time and (*b*) the filtration cycle with the existing pump for the maximum daily capacity, if the time for removing the cake and reassembling the press is 20 minutes. The cake is not washed.

5. A rotary filter, operating at 2 r.p.m., filters 100 g.p.m. Operating under the same vacuum and neglecting the resistance of the filter cloth, at what speed must the filter be operated to give a filtration rate of 200 g.p.m.?

6. A slurry is filtered in a plate and frame press containing 12 frames, each 1 ft square and 1 in. thick. During the first three minutes, the filtration pressure is slowly raised to the final value of 60 lb/in.$^2$ and, during this period, the rate of filtration is maintained constant. After the initial period, filtration is carried out at constant pressure and the cakes are completely formed in a further 15 minutes. The cakes are then washed at 40 lb/in.$^2$ for 10 minutes, using "thorough washing." What is the volume of filtrate collected per cycle and how much wash water is used?

A sample of the slurry had previously been tested, using a vacuum leaf filter of $\frac{1}{2}$ ft$^2$ filtering surface and a vacuum of 20 in. mercury. The volume of filtrate collected in the first 5 minutes was 250 cc and, after a further 5 minutes, an additional 150 cc was collected. Assume the cake to be incompressible and the cloth resistance to be the same in the leaf as in the filter press.

7. A sludge is filtered in a plate and frame press fitted with 1 in. frames. For the first ten minutes the slurry pump runs at maximum capacity. During this period the pressure rises to 60 lb/in.$^2$ and a quarter of the total filtrate is obtained. The filtration takes a further hour to complete at constant pressure and 15 minutes are required for emptying and resetting the press.

It is found that, if the cloths are precoated with filter aid to a depth of $\frac{1}{16}$ in., the cloth resistance is reduced to a quarter of its former value. What will be the increase in the overall throughput of the press if the precoat can be applied in three minutes?

8. Filtration is carried out in a plate and frame filter press, with 20 frames 1 ft square and 2 in. thick, and the rate of filtration is maintained constant for the first five minutes. During this period, the pressure is raised to 50 lb/in.$^2$, and one-quarter of the total filtrate per cycle is obtained. At the end of the constant rate period, filtration is continued at a constant pressure of 50 lb/in.$^2$ for a further 30 minutes, after which the frames are full. The total volume of filtrate per cycle is 150 gallons and dismantling and refitting of the press takes 15 minutes.

It is decided to use a rotary drum filter, 5 ft long and $7\frac{1}{4}$ ft in diameter, in place of the filter press. Assuming that the resistance of the cloth is the same in the two plants and that the filter cake is incompressible, calculate the speed of rotation of the drum which will result in the same overall rate of filtration as was obtained with the filter press. The filtration in the rotary filter is carried out at a constant pressure difference of 10 lb/in.$^2$, and the filter operates with 25% of the drum submerged in the slurry at any instant.

9. It is required to filter a certain slurry to produce 500 gallons of filtrate per working day of eight hours. The process is carried out in a plate and frame filter press with 18 in. square frames and a working pressure of 50 lb/in.$^2$ gauge. The pressure is built up slowly over a period of five minutes and, during this period, the rate of filtration is maintained constant.

When a sample of the slurry was filtered, using a vacuum of 20 in. mercury, on a single leaf filter of filtering area 72 in.$^2$, 400 cc of filtrate were collected in the first five minutes of filtration and a further 400 cc were collected during the following ten minutes. Assuming that the dismantling of the filter press, the removal of the cakes and the setting up again of the press takes an overall time of five minutes, plus an additional three minutes for each cake produced, what is the minimum number of frames that need be employed? Take the resistance of the filter cloth to be the same in the laboratory tests as on the plant.

10. If a centrifuge is 3 ft diameter and rotates at 1000 r.p.m., what speed should a laboratory centrifuge of 6 in. diameter be run if it is to duplicate plant conditions?

11. What is the maximum safe speed of rotation of a phosphor-bronze centrifuge basket, 12 in. diameter and $\frac{3}{16}$ in. thick, when it contains a liquid of specific gravity unity forming a layer 3 in. thick at the walls? Take the specific gravity of phosphor-bronze as 8·9 and the safe working stress as 8000 lb/in.$^2$

12. When an aqueous slurry is filtered in a plate and frame press, fitted with two 2 in. thick frames each 6 in. square at 50 lb/in.$^2$ gauge pressure, the frames are filled in 1 hour. The liquid in the slurry has the same density as water. The slurry is then filtered in a perforate basket centrifuge with a basket 12 in. diameter and 8 in. deep. If the radius of the inner surface of the slurry is maintained constant at 3 in. and the speed of rotation is 4000 r.p.m., how long will it take to produce as much filtrate as was obtained from a single cycle of operations with the filter press?

Assume that the filter cake is incompressible and that the resistance of the cloth is equivalent to $\frac{1}{8}$ in. of cake in both cases.

13. A centrifuge with a phosphor bronze basket 15 in. diameter is to be run at 2000 r.p.m., with a 4 in. layer of solids of bulk density 2·0 g/cc at the walls. What should be the thickness of the walls of the basket if the perforations are so small that they have a negligible effect on the strength?

Density of phosphor bronze = 8·9 g/cc
Maximum safe stress for phosphor bronze = 8000 lb/in.$^2$

14. A finely ground mixture of galena and limestone in the proportion of 1 to 4 by weight, is subjected to elutriation by an upwards current of water flowing at 0·5 cm/sec. Assuming that the size distribution for each material is the same, and as shown by the following table, estimate the percentage of galena in the material carried away and in the material left behind. Take the absolute viscosity of water as 0·01 poise and use Stokes' equation.

| Diameter in microns | 20 | 30 | 40 | 50 | 60 | 70 | 80 | 100 |
|---|---|---|---|---|---|---|---|---|
| Percentage weight of undersize | 15 | 28 | 48 | 54 | 64 | 72 | 78 | 88 |

Specific gravity of galena = 7·5; specific gravity of limestone = 2·7.

15. Calculate the terminal velocity of a steel ball, 2 mm diameter (density 7·87 gm per cc) in oil (density 0·9 gm per cc, viscosity 0·5 poise).

16. What will be the settling velocity of a spherical steel particle, 0·015 in. diameter, in an oil of specific gravity 0·82 and viscosity 10 centipoises? The specific gravity of steel is 7·87.

17. What are the settling velocities of mica plates, $\frac{1}{32}$ in. thick and ranging in area from 0·01 to 1 in.$^2$, in an oil of specific gravity 0·82 and viscosity 10 centipoises. The specific gravity of mica is 3·0.

18. A material of specific gravity 2·5 is fed to a size separation plant where the separating fluid is water which rises with a velocity of 4 f.p.s. The upward vertical component of the velocity of the particles is 20 f.p.s. How far will an approximately spherical particle, $\frac{1}{4}$ in. diameter, rise relative to the walls of the plant before it comes to rest in the fluid?

19. Oil, of specific gravity 0·9 and viscosity 3 centipoises, passes vertically upwards through a bed of catalyst consisting of approximately spherical particles of diameter 0·1 millimetre and specific gravity 2·6. At approximately what mass rate of flow per unit area of bed will (*a*) fluidisation, and (*b*) transport of particles, occur?

20. A sample of dust from the air in a factory has been collected on a glass slide. If the dust on the slide was deposited from one cubic centimetre of air, estimate the weight of dust in grams per cubic metre of air in the factory, given the number of particles in the various size ranges to be as shown in the following table.

| Size range in microns: | 0–1 | 1–2 | 2–4 | 4–6 | 6–10 | 10–14 |
|---|---|---|---|---|---|---|
| Number of particles: | 2000 | 1000 | 500 | 200 | 100 | 40 |

Assume the specific gravity of the dust to be 2·6, and make an appropriate allowance for particle shape.

21. The size distribution by weight of the dust carried in a gas is given in the following table, together with the efficiency of collection over each size range.

| Size range, microns | 0–5 | 5–10 | 10–20 | 20–40 | 40–80 | 80–160 |
|---|---|---|---|---|---|---|
| Per cent weight | 10 | 15 | 35 | 20 | 10 | 10 |
| Per cent efficiency | 20 | 40 | 80 | 90 | 95 | 100 |

Calculate the overall efficiency of the collector and the percentage by weight of the emitted dust which is smaller than 20 microns in diameter. If the dust burden is 0·5 gm/ft$^3$ at entry and the gas flow 40,000 ft$^3$/min, calculate the weight of dust emitted in tons per day.

22. The collection efficiency of a cyclone is 45% over the size range 0 to 5 microns, 80% over the size range 5 to 10 microns, and 96% for particles exceeding 10 microns. Calculate the efficiency of collection for the following dust:—

Weight distribution: 50% 0 to 5 microns.
30% 5 to 10 microns.
20% above 10 microns.

23. Ten tons per hour of dry sea shore sand, containing 1% by weight of salt, is to be washed with 10 tons per hour of fresh water running countercurrent to the sand through two classifiers in series. Assume that perfect mixing of the sand and water occurs in each classifier

and that the sand discharged from each classifier contains one part of water for every two of sand (by weight). If the washed sand is dried in a kiln dryer, what percentage of salt will it retain?

What wash rate would be required in a single classifier in order to wash the sand equally well?

24. Caustic soda is manufactured by the lime-soda process according to the following equation—

$$Na_2CO_3 + Ca(OH)_2 = 2NaOH + CaCO_3$$

A solution of sodium carbonate in water (25 tons/day $Na_2CO_3$) is treated with the theoretical requirement of lime and, after the reaction is complete, the $CaCO_3$ sludge, containing by weight one part of $CaCO_3$ per nine parts of water is fed continuously to three thickeners in series and is washed countercurrently. Calculate the necessary rate of feed of neutral water to the thickeners, so that the calcium carbonate, on drying, contains only 1% of sodium hydroxide. The solid discharged from each thickener contains one part by weight of calcium carbonate to three of water.

25. How many stages are required for 98% extraction of a material containing 18% of extractable matter (having a sp. gr. of 2·7) and which requires 200 volumes of liquid per 100 volumes of solid for it to be capable of being pumped to the next stage. The strong solution is to have a concentration of 100 grams per litre.

26. Soda ash is mixed with lime and the liquor from the second of three thickeners and passes to the first thickener where separation is effected. The quantity of this caustic solution leaving the first thickener is such as to yield 10 tons of caustic soda per day of 24 hours. The solution contains 0·095 ton of caustic soda per ton of water, whilst the sludge leaving each of the thickeners consists of one part of solids to one of liquid.

Determine:

(*a*) the weight of solids in the sludge;

(*b*) the weight of water admitted to the third thickener;

(*c*) the percentages of caustic soda in the sludges leaving the respective thickeners.

27. Seeds, containing 20% by weight of oil, are extracted in a countercurrent plant and 90% of the oil is recovered in a solution containing 50% by weight of oil. If the seeds are extracted with fresh solvent and 1 pound of solution is removed in the underflow in association with every two pounds of insoluble matter, how many ideal stages are required? Use a triangular diagram.

28. It is desired to recover precipitated chalk from the causticising of soda ash. After decanting the liquor from the precipitators the sludge has the composition 5% $CaCO_3$, 0·1% NaOH and the balance water.

One thousand tons per day of this sludge is fed to two thickeners where it is washed with 200 tons per day of neutral water. The pulp removed from the bottom of the thickeners contains 4 tons of water per ton of chalk. The pulp from the last thickener is taken to a rotary filter and concentrated to 50% solids and the filtrate is returned to the system as wash water.

Calculate the net percentage of $CaCO_3$ in the product after drying.

29. Barium carbonate is to be made by the interaction of sodium carbonate and barium sulphide. The quantities that are fed to the reaction agitators per 24 hours are as follows: 40,000 lb of barium sulphide dissolved in 60 tons of water together with the theoretically necessary amount of sodium carbonate.

There are three thickeners in series, run on a countercurrent decantation system. Overflow from the second thickener goes to the agitators and overflow from the first thickener is to be 10% sodium sulphide. Sludge from all thickeners carries two parts water to one part barium carbonate (w/w).

How much sodium sulphide will remain in the dried barium carbonate precipitate?

30. In the production of caustic soda by the action of calcium hydroxide on sodium carbonate, 100 tons per day of sodium carbonate per day is treated with the theoretical quantity of lime. The sodium carbonate is made up as a 20% solution. The material from the extractors is fed to a countercurrent washing system where it is treated with 200 tons per day of clean water. The washing thickeners are so arranged that the ratio of the volume of liquid discharged in the liquid offtake to that discharged with the solid is the same in all the thickeners and is equal to 4·0. How many thickeners must be arranged in series so that not more than 1% of the sodium hydroxide discharged with the solid from the first thickener is wasted?

31. A plant produces 100 tons per day of titanium dioxide pigment which must be 99·9% pure when dried. The pigment is produced by precipitation and the material, as prepared, is contaminated with 1 ton of salt solution containing 0·55 ton of salt per ton of pigment. The material is washed countercurrently with water in a number of thickeners arranged in series. How many thickeners will be required if water is added at the rate of 200 tons per day and the solid discharged from each thickener removes ½ ton of solvent per ton of pigment?

What will be the required number of thickeners if the amount of solution removed in association with the pigment varies in the following way with the concentration of the solution in the thickener?

| *Concentration (solute/solution)* | *Solution/unit mass of pigment* |
|---|---|
| 0 | 0·30 |
| 0·1 | 0·32 |
| 0·2 | 0·34 |
| 0·3 | 0·36 |
| 0·4 | 0·38 |
| 0·5 | 0·40 |

The concentrated wash liquor is mixed with the material fed to the first thickener.

32. A liquor of four components, *A*, *B*, *C* and *D*, with 0·3 mol fraction each of *A*, *B* and *C*, is to be continuously fractionated to give a top product of 0·9 mol fraction *A* and 0·1 mol fraction *B*. The bottoms are to contain not more than 0·5 mol fraction *A*. Estimate the minimum reflux ratio required for this separation, if the relative volatility of *A* to *B* is 2·0.

33. During the batch distillation of a binary mixture in a packed column the product had 0·65 mol fraction of the more volatile component when the concentration in the still was 0·40 mol fraction. If the reflux ratio in use was 20 : 1, and the vapour composition $y$ is related to the liquor composition $x$ by the equation $y = 1{\cdot}035x$, over the range of concentration concerned, determine the number of ideal plates represented by the column ($x$ and $y$ are in mol fractions).

34. A mixture of water and ethyl alcohol containing 0·16 mol fraction alcohol is continuously distilled in a plate fractionating column to give a product containing 0·77 mol fraction alcohol and a waste of 0·02 mol fraction alcohol. It is proposed to withdraw 25% of the alcohol in the entering stream as a side stream with a mol fraction of 0·50 alcohol.

Determine the number of theoretical plates required and the plate from which the side stream should be withdrawn if the feed is liquor at the boiling point and a reflux ratio of 2 is used.

35. The mol fractions of a mixture to be fed to a continuous distillation column is approximately phenol 0·35, *o*-Cresol 0·15, *m*-Cresol 0·30 and xylenols 0·20. It is hoped to obtain a mol fraction in the product of phenol 0·952, of *o*-Cresol 0·0474 and of *m*-Cresol 0·0006. If the volatility relative to *o*-Cresol of phenol is 1·26 and of *m*-Cresol 0·70, estimate how many theoretical plates would be required at total reflux.

36. A continuous fractionating column, operating at atmospheric pressure, is to be designed to separate a mixture containing 15·67% $CS_2$ and 84·33% $CCl_4$ into an overhead product containing 91% $CS_2$ and a waste of 97·3% $CCl_4$ (all weight per cent). Assume a plate efficiency of 70% and a reflux of 3·16 mols per mol of product.

Using the data below, determine the number of plates required.

Feed enters at 15°C with a specific heat of 0·4 and boiling-point of 63°C.

Latent heat of $CS_2$ and $CCl_4$ is 6200 cal/gm-mol.

Mols per cent $CS_2$ in vapour:

0, 8·23, 15·55, 26·6, 33·2, 49·5, 63·4, 74·7, 82·9, 87·8, 93·2.

Mols per cent $CS_2$ in liquor:

0, 2·96, 6·15, 11·06, 14·35, 25·85, 39·0, 53·18, 66·30, 75·75, 86·04.

37. A batch fractionation is carried out in a small column which has the separating power of 6 theoretical plates. The mixture consists of benzene and toluene with 0·60 mol fraction of benzene. A distillate is required, of constant composition, of 0·98 mol fraction benzene, and the operation is discontinued when 83% of the benzene charged has been removed as distillate. Estimate the reflux ratio needed at the start and finish of the distillation, if the relative volatility of benzene to toluene is taken as 2·46.

38. In a distillation unit separating an ideal binary mixture, show how the thermodynamic efficiency may be obtained by considering the minimum work required for separation per mole of feed and the available work in the heat put into the still and removed from the condenser.

An ideal mixture contains 0·2 mol fraction of the more volatile component. This is to be separated at a minimum reflux ratio of 3·67 under conditions where the boiler temperature is 692°R, the still top temperature 636°R and the cooling water temperature 546°R. Under these conditions, what will be the thermodynamic efficiency of the unit?

It may be assumed that complete separation is obtained and that the feed is liquid at its boiling point of 677°R; the latent heat may be taken as 13,400 B.Th.U./lb-mol.

Indicate by a simple diagram a practical system which gives improved efficiency.

39. A continuous fractionating column is required to separate a mixture containing 0·695 mol fraction *n*-heptane ($C_7H_{16}$) and 0·305 mol fraction *n*-octane ($C_8H_{18}$) into products of 99 mol % purity. The column is to operate at a pressure of 1 atmosphere with a vapour velocity of 2ft/sec. The feed is all liquid at its boiling point, and is supplied to the column at 10,000 lb/hr. The boiling point at the top of the column may be taken as 99°C, and the equilibrium data are—

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| $y$ = mol fraction heptane in vapour | 0·96 | 0·91 | 0·83 | 0·74 | 0·65 | 0·5 | 0·37 | 0·24 |
| $x$ = mol fraction heptane in liquid | 0·92 | 0·82 | 0·69 | 0·57 | 0·46 | 0·32 | 0·22 | 0·13 |

Determine:

(*a*) The minimum reflux ratio that will be required.

(*b*) What diameter column would be required if the reflux used were twice the minimum possible?

40. The vapour pressures of chlorobenzene and water are as follows—

| Vapour pressure, mm mercury | 100 | 50 | 30 | 20 |
|---|---|---|---|---|
| Temperatures °C | | | | |
| Chlorobenzene | 70·4 | 53·7 | 42·7 | 34·5 |
| Water | 51·7 | 38·5 | 29·9 | 22·5 |

A still is operated at a pressure of 130 mm absolute, steam being blown continuously into it. Estimate the temperature of the boiling liquid and the composition of the distillate if liquid water is present in the still.

41. The following figures represent the equilibrium conditions in mol fraction of benzene in benzene-toluene mixtures at their boiling point.

| *Liquid* | *Vapour* |
|---|---|
| 0·51 | 0·72 |
| 0·38 | 0·60 |
| 0·26 | 0·45 |
| 0·15 | 0·30 |

If the liquid compositions on four adjacent plates in a column were 0·18, 0·28, 0·41 and 0·57 under conditions of total reflux, determine the plate efficiencies.

42. A continuous rectifying column treats a mixture consisting of 40% of benzene by weight and 60% of toluene at the rate of 30,000 lb/hr, and separates it into a product containing 97% of benzene and a liquid containing 98% toluene. The feed is liquid at its boiling point.

(*a*) Calculate the weights of distillate and waste liquor per hour.

(*b*) If a reflux ratio of 3·5 to 1 is employed, how many plates are required in the rectifying part of the column?

(*c*) What is the actual number of plates if the plate-efficiency is 60%?

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Mol fraction of benzene in liquid | 0·1 | 0·2 | 0·3 | 0·4 | 0·5 | 0·6 | 0·7 | 0·8 | 0·9 |
| Mol fraction of benzene in vapour | 0·22 | 0·38 | 0·51 | 0·63 | 0·7 | 0·78 | 0·85 | 0·91 | 0·96 |

43. The accompanying table gives the relationship between the mol fraction of carbon disulphide in the liquid and in the vapour evolved from the mixture during the distillation of a carbon disulphide-carbon tetrachloride mixture.

*Table*

| | | | | | | |
|---|---|---|---|---|---|---|
| *x* | 0 | 0·20 | 0·40 | 0·60 | 0·80 | 1·00 |
| *y* | 0 | 0·445 | 0·65 | 0·795 | 0·91 | 1·00 |

Determine graphically the theoretical number of plates required for the rectifying and stripping portions of the column, using the following data:

Reflux ratio, 3 to 1.
Slope of fractionating line, 1·4.
Purity of product, 99%.
Percentage carbon disulphide in waste liquors, 1%.

What is the minimum slope of the rectifying line in this case?

44. A fractionating column is required to distil a liquid containing 25% benzene and 75% toluene by weight, so as to give a product of 90% benzene. A reflux ratio of 3·5 is to be used, and the feed will enter at its boiling point.

If the plates used are 100% efficient, calculate by the LEWIS-SOREL method the composition of liquid on the third plate, and by MCCABE and THIELE's method estimate the number of plates required.

45. A 50 mol % mixture of benzene and toluene is fractionated in a batch still which has the separating power of 8 theoretical plates. It is proposed to obtain a constant quality product with a mol % benzene of 95, and to continue the distillation till the still has a content of 10% of benzene.

What will be the reflux ratios used in the process and how much of the distillate will be obtained at each value of the reflux ratio?

46. The vapour composition on a plate of a distillation column is—

| | | |
|---|---|---|
| $C_1$ | 0·025 mol fraction | 36·5 rel. volatility |
| $C_2$ | 0·205 | 7·4 |
| *i*-$C_3$ | 0·210 | 3·0 |
| *n*-$C_3$ | 0·465 | 2·7 |
| *i*-$C_4$ | 0·045 | 1·3 |
| *n*-$C_4$ | 0·050 | 1·0 |

What will be the composition of the liquid on the plate if it is in equilibrium with the vapour?

47. A liquor of 0·30 mol fraction of benzene and the rest toluene is fed to a continuous still to give a top product of 0·90 mol fraction benzene and a bottom product of 0·95 mol fraction toluene.

If the reflux ratio is 5·0, how many plates are required

(*a*) if the feed is saturated vapour?
(*b*) if the feed is liquid at 10°C?

48. A mixture of alcohol and water with 0·45 mol fraction of alcohol is to be continuously distilled in a column so as to give a top product of 0·825 mol fraction alcohol and a liquor at the bottom with 0·05 mol fraction alcohol.

How many theoretical plates are required if the reflux ratio used is 3? Indicate on the diagram what is meant by the Murphree plate efficiency.

49. It is desired to separate 1 ton/hr of an ammonia solution containing 30% $NH_3$ by weight into 99·5% liquid $NH_3$ and a residual weak solution containing 10% $NH_3$. Assuming the feed to be at its boiling point, a column pressure of 10 atm. abs., a plate efficiency of 60% and that 8% excess over minimum reflux requirements is used, how many plates must be used in the column and how much heat is removed in the condenser and added in the boiler?

50. A $P - x$ diagram has been determinated at 184°C for a binary mixture. The vapour pressures of the pure components are $P_A{}^0 = 300$ mm and $P_B{}^0 = 400$ mm, and for $x_A = 0{\cdot}4$, $P_A = 156$ and $P_B = 288$.

Calculate the temperature and the value of $y_A$ for a liquid composition of $x_A = 0{\cdot}4$, if a distillation is carried out by varying the temperature for a constant total pressure of 400 mm.

Assume the activity coefficients to be independent of temperature.

The following vapour pressure data are given:

| Temp. °C | 178 | 180 | 182 | 184 | 186 |
|---|---|---|---|---|---|
| $P_A{}^\circ$ | 245 | 263 | 281 | 300 | 319 |
| $P_B{}^\circ$ | 320 | 345 | 371 | 400 | 430 |

51. A mixture of 60 mol % benzene, 30% of toluene and 10% xylene is run into a batch still. If the top product is to be 99% benzene, determine

(*a*) the liquid composition on each plate at total reflux,
(*b*) the composition on the 2nd and 4th plates for $R = 1{\cdot}5$,
(*c*) as for (*b*) but $R = 3$,
(*d*) as for (*c*) but $R = 5$,
(*e*) as for (*d*) but $R = 8$ and for the condition when the mol % benzene in the still is 10.
(*f*) as for (*e*) but with $R = 5$.

Take the relative volatility of benzene to toluene as 2·4, and xylene to toluene as 0·43.

52. A continuous still is fed with a mixture of mol fraction 0·5 of the more volatile component, and gives a top product of 0·9 mol fraction more volatile component and a bottom product of 0·10 mol fraction.

If the still operates with an $L_n/D$ ratio of 3·5 : 1, calculate by SOREL's method the composition of the liquid on the third theoretical plate from the top,

(*a*) for benzene-toluene and
(*b*) for *n*-heptane-toluene.

53. 60,000 lb/hr of *n*-propanol is obtained off the top of a distillation column working at 15 lb/in.²G. The propanol is totally condensed and part taken for product and part returned for reflux. Under these conditions the boiling point is 244°F and the liquid density 48·5 lb/ft³.

Determine the diameter and plate spacing based on entrainment and estimate the height of liquor in the downcomer. The liquor flow down may be taken as 40,000 lb/hr.

What size do you suggest for the downcomer, the weir, the liquid seal and the area of the risers to the bubble caps? The $F_w$-factor for the height over the weir may be taken as 1.

54. Determine the minimum reflux ratio by the following two methods for the three systems given below.

(*a*) Using Fenske's equation.

(*b*) Using Colburn's rigorous method.

1. $C_6$ (0·60 mol fraction) $C_7$ (0·30) $C_8$ (0·10) to give a product of 0·99 mol fraction $C_6$.

| | | | *Mol Frac.* | *Rel. Vol.* $\alpha$ | $x_d$ |
|---|---|---|---|---|---|
| 2. | Components | *c* | 0·3 | 2 | 1·0 |
| | | *d* | 0·3 | 1 | — |
| | | *e* | 0·4 | $\frac{1}{2}$ | — |
| 3. | Components | *c* | 0·25 | 2 | 1·0 |
| | | *d* | 0·25 | 1 | — |
| | | *e* | 0·25 | $\frac{1}{2}$ | — |
| | | *f* | 0·25 | $\frac{1}{4}$ | — |

55. A liquor consisting of phenol and cresols with some xylenols is fractionated to give a top product of 95·3 mol % phenol. The compositions of the top product and of the phenol in the bottoms are given. A reflux ratio of 10 will be used.

(*a*) Complete the material balance over the still for a feed of 100 mols.

(*b*) Calculate the composition on the second plate from the top.

(*c*) Calculate the composition on the second plate from the bottom.

(*d*) Calculate the minimum reflux ratio by Underwood's equation and by Colburn's approximation.

| | *Compositions in mol %* | | |
|---|---|---|---|
| | *Feed* | *Top* | *Bottom* |
| phenol | 35 | 95·3 | 5·24 |
| *o*-cresol | 15 | 4·55 | |
| *m*-cresol | 30 | 0·15 | |
| xylenols | 20 | | |
| | 100 | 100 | 100 |

Heavy key is *m*-cresol, light key is phenol.

56. A continuous fractionating column is to be designed to separate 20,000 lb/hr of a mixture of 60% toluene and 40% benzene, so as to give an overhead of 97% benzene and a waste of 98% toluene by weight.

A reflux ratio of 3·5 mols of reflux per mol of product is to be used and the molar latent heat of benzene and toluene may be taken as 7240 cals per gram mol.

Calculate

(*a*) The weight of product and waste per hour.

(*b*) No. of theoretical plates and position of feed if the feed is liquid at 20°C, of sp. heat 0·44.

(*c*) How much steam at 20 lb/in.$^2$-G. is required in the still.

(*d*) What will be the required diameter of the column if it operates at atmospheric pressure and a vapour velocity of 3 ft/sec.

(*e*) If the vapour velocity is to be 2·5 ft/sec, based on free area of column, the necessary diameter of the column.

(*f*) The minimum possible reflux ratio and what is the minimum number of plates for a feed entering at its boiling point.

57. For a system that obeys RAOULT's law show that the relative volatility $\alpha_{AB}$ is $P_A{}^0/P_B{}^0$, where $P_A{}^\circ$ and $P_B{}^\circ$ are the vapour pressures of the components *A* and *B* at the given temperature.

From the vapour pressure curves of benzene, toluene, ethyl benzene and of ortho-, meta- and para-xylenes, obtain a plot of the volatilities of each of the materials relative to meta-xylene over the range of 70–160°C.

58. A still has a liquor composition of ortho-xylene 10%, meta-xylene 65%, para-xylene 17%, benzene 4% and ethyl benzene 4%. How many plates at total reflux are required to give a product of 80% meta-xylene, and 14% para-xylene? The data are given as weight per cent.

59. The vapour pressure of *n*-pentane and of *n*-hexane is given in the table below.

| Pressure in mm of Hg | | 10 | 20 | 40 | 60 | 100 | 200 | 400 | 760 |
|---|---|---|---|---|---|---|---|---|---|
| $C_5H_{12}$ | Temp. in °C | −50·1 | −40·2 | −29·2 | −22·2 | −12·6 | 1·9 | 18·5 | 36·1 |
| $C_6H_{14}$ | | −25 | −14·1 | −2·3 | 5·4 | 15·8 | 31·6 | 49·6 | 68·7 |

Equilibrium Curve:

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| $x =$ | 0·1 | 0·2 | 0·3 | 0·4 | 0·5 | 0·6 | 0·7 | 0·8 | 0·9 |
| $y =$ | 0·21 | 0·41 | 0·54 | 0·66 | 0·745 | 0·82 | 0·875 | 0·925 | 0·975 |

(*a*) Determine the relative volatility of pentane to hexane at temperatures of 0, 20 and 40°C.

(*b*) A mixture containing 0·52 mol fraction pentane is to be continuously distilled to give a top product of 0·95 mol fraction pentane and a bottom of 0·1 mol fraction pentane.

Determine the minimum number of plates and the number of plates at total reflux by the graphical McCABE-THIELE method and analytically by using the relative volatility method.

(*c*) Using the conditions as in (*b*), determine the liquid composition on the second plate from the top by LEWIS's method, if a reflux ratio of 2 is used.

(*d*) Using the conditions as in (*b*), determine by McCABE-THIELE method the total number of plates required, and the position of the feed.

It may be assumed that the feed is all liquid at the boiling point.

60. Some experiments are made on the absorption of carbon dioxide from a carbon dioxide-air mixture in 2·5 normal caustic soda, using a 10 in. diameter tower packed to a height of 10 ft with ¾ in. Raschig rings.

In one experiment at atmospheric pressure, the results obtained were—

Gas rate *G*′: 263 lb/hr-ft². Liquid *L*′: 3050 lb/hr-ft².

The carbon dioxide in the inlet gas is 315 parts per million and in the exit gas 31 parts per million.

What is the value of the overall gas transfer coefficient $K_Ga$?

It was found that the results could be expressed by a relation

$$K_Ga = \beta' \frac{G'^{0\cdot35}L'^{0\cdot20}}{P^{\frac{1}{2}}}$$

where $\beta'$ is a constant and *P* the pressure.

What conclusion would you draw as to the relative magnitudes of the film resistances?

61. An acetone-air mixture containing 0·015 mol fraction of acetone has the mol fraction reduced to $\frac{1}{100}$ of this value by countercurrent absorption with water in a packed tower. The gas flow rate *G* is 800 lb/hr-ft² of air and the water entering is 1250 lb/hr-ft². For this system, HENRY's law holds and $y_e = 1{\cdot}75x$, where $y_e$ is the mol fraction of acetone in the vapour in equilibrium with a mol fraction *x* in the liquid. How many overall transfer units are required?

62. An oil containing 2·55 mol % of a hydrocarbon is stripped by running the oil down a column up which live steam is passed, so that 4 mols of steam are used per 100 mols of oil stripped. Determine the number of theoretical plates required to reduce the hydrocarbon content to 0·05 mol %, assuming that the oil is non-volatile. The vapour-liquid relation of the hydrocarbon in the oil is given by $y_e = 33x$, where $y_e$ is the mol fraction in the vapour and $x$ the mol fraction in the liquid. The temperature is maintained constant by internal heating, so that the steam does not condense in the tower.

63. Gas, from a petroleum distillation column, has its concentration of $H_2S$ reduced from 0·03 mols $H_2S$/mol of inert hydrocarbon gas to 1/10th of this value, by scrubbing with a triethanolamine-water solvent in a countercurrent tower, operating at 80°F and at atmospheric pressure.

$H_2S$ is soluble in such a solution and the equilibrium relation may be taken as $Y = 2X$, where $Y$ is mols of $H_2S$/mol of inert gas and $X$ is mols of $H_2S$/mol of solvent.

The solvent enters the tower free of $H_2S$ and leaves containing 0·013 mols of $H_2S$/mol of solvent. If the flow of inert hydrocarbon gas is 10 lb-mol/hr-ft² of tower cross-section and the gas-phase resistance controls the process, calculate

(*a*) the height of the absorber necessary;

(*b*) the number of transfer units required.

The overall coefficient for absorption $K_G''a$ may be taken as 8 lb-mols/hr-ft³ of tower volume (unit driving force in $Y$).

64. It is known that the overall liquid transfer coefficient $K_La$ for absorption of $CO_2$ in water in a column is 11·5 lb-mols/hr-ft³ (lb-mols/ft³). By assuming an expression for the absorption of $NH_3$ in water at the same liquor rate and varying gas rates, derive an expression for the overall liquid film coefficient $K_La$ for absorption of $SO_2$ in water in the same apparatus at the same water rate but varying gas rates. The diffusivities of $SO_2$ and $NH_3$ in air at 0°C are 0·103 and 0·170 cm²/sec. $SO_2$ dissolves in water, so that HENRY's constant $\mathscr{H}$ is equal to 7·7 atmospheres/(lb-mol/ft³). All data are expressed for the same temperature.

65. A packed tower is used for absorbing sulphur dioxide from air by means of a N/2 caustic soda solution. At an air flow of 1570 lb per hour per ft², corresponding to a Reynolds number of 5160, the friction factor $R/\rho u^2$ is found to be 0·0200.

Calculate the mass transfer coefficient in lbs $SO_2$/hr-ft²-atm. under these conditions if the tower is at atmospheric pressure. At the temperature of absorption the following values may be taken.

Diffusion coefficient $SO_2$ = 0·116 cm²/sec.
Viscosity of gas = 0·00018 poise.
Density of gas stream = $1{\cdot}154 \times 10^{-3}$ gm/cc.

66. In an absorption tower, ammonia is being absorbed from air at atmospheric pressure by acetic acid. The flow rate of 1550 lb/hr-ft² in an experiment corresponds to a Reynolds number of 5100 and hence a friction factor $R/\rho u^2$ of 0·0199. At the temperature of absorption the viscosity of the gas stream ($\mu$) is 0·00018, the density ($\rho$) is 0·001154 and the diffusion coefficient of ammonia in air ($D$) is 0·196. All these figures are expressed in c.g.s. units. Determine the mass transfer coefficient through the gas film in lb/hr-ft²-atm.

67. A soluble gas is absorbed from a dilute gas-air mixture by countercurrent scrubbing with a solvent in a packed tower. If the liquor led to the top of the tower contains no solute, show that the number of transfer units required is given by:

$$N = \frac{1}{\left[1 - \frac{m\,.\,G_m}{L_m}\right]} \ln \left[\left(1 - \frac{m\,.\,G_m}{L_m}\right)\frac{y_1}{y_2} + \frac{m\,.\,G_m}{L_m}\right]$$

where $G_m$ and $L_m$ are the flow rates of the gas and liquid in moles per hr per ft² of tower area, and $y_1$ and $y_2$ the mol fraction of the gas at the inlet and outlet of the column. The equilibrium relation between the gas and liquid is represented by a straight line with the equation $y_e = mx$, where $y_e$ is the mol fraction in the gas in equilibrium with $x$ mol fraction in the liquid.

In a given process, it is desired to recover 90% of the solute by using 50% more liquid than the minimum necessary. If the H.T.U. of the proposed tower is 2 ft, what height of packing will be required?

68. A paraffin hydrocarbon of molecular weight 114, at a temperature of 100°C, is to be separated from admixture with a non-volatile organic compound of molecular weight 135 by stripping with steam. The liquor contains 8% of the paraffin by weight and this is to be reduced to 0·08% using an upward flow of steam saturated at 100°C. If three times the minimum amount of steam is used, how many theoretical stages will be required?

The vapour pressure of the paraffin at 100°C is 400 mm of mercury and the process takes place at atmospheric pressure. It may be assumed that the system obeys RAOULT's law.

69. Acetone is to be recovered from a 5% acetone-air mixture by scrubbing with water in a packed tower using countercurrent flow. The liquid rate is 650 lb/hr-ft$^2$ and the gas rate is 390 lb/hr-ft$^2$.

The overall absorption coefficient $K_G$ may be taken as 3·7 lb-mols/hr-ft$^2$ atmosphere partial pressure difference, and the gas film resistance controls the process.

What should be the height of the tower to remove 98% of the acetone? The equilibrium data for the mixture are—

| | | | | |
|---|---|---|---|---|
| Mol fraction acetone in gas | 0·0099 | 0·0196 | 0·0361 | 0·0400 |
| Mol fraction acetone in liquid | 0·0076 | 0·0156 | 0·0306 | 0·0333 |

70. Ammonia is to be removed from 10% ammonia-air mixture by countercurrent scrubbing with water in a packed tower at 20°C so that 99% of the ammonia is removed when working at a total pressure of 760 mm of mercury.

If the gas rate is 735 lb/hr-ft$^2$ of tower cross-section and the liquid rate is 500 lb/hr-ft$^2$, find the necessary height of the tower if the absorption coefficient is $K_Ga = 23{\cdot}6$ lb-mols/ft$^3$-hr-atmosphere partial pressure difference. The equilibrium data are—

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Mols $NH_3$/mol water | 0·021 | 0·031 | 0·042 | 0·053 | 0·079 | 0·106 | 0·159 |
| Partial pressure $NH_3$ in mm of Hg | 12·0 | 18·2 | 24·9 | 31·7 | 50·0 | 69·6 | 114·0 |

71. Sulphur dioxide is recovered from a smelter gas containing 3·5% by volume of $SO_2$, by scrubbing with water in a countercurrent absorption tower. The gas is fed into the bottom of the tower, and in the exit gas from the top the $SO_2$ exerts a partial pressure of 8·53 mm of Hg. The water fed to the top of the tower is free from $SO_2$, and the exit liquor from the base contains 0·001145 mols $SO_2$ per mol water. The process takes place at 68°F, at which the vapour pressure of water is 17·5 mm of Hg. The water rate is 3420 lb-mols per hour.

If the area of the tower is 20 ft$^2$ and the overall coefficient of absorption for these conditions $K_L''a$ is 42 lb-mols $SO_2$/hr (ft$^3$) (mols of $SO_2$ per mol $H_2O$), calculate the necessary height of the column.

The equilibrium data for $SO_2$ and water at 68°F are—

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Mols $SO_2$ per 1000 mols $H_2O$ | 0·056 | 0·14 | 0·28 | 0·42 | 0·56 | 0·84 | 1·405 |
| Mols $SO_2$ per 1000 mols inert gas | 0·7 | 1·6 | 4·3 | 7·9 | 11·6 | 19·4 | 36·3 |

72. Ammonia is removed from a 10% ammonia-air mixture by scrubbing with water in a packed tower, so that 99·9% of the ammonia is removed. What is the required height of tower?

| | |
|---|---|
| Entering gas | 900 lb/hr-ft$^2$ |
| Water rate | 720 lb/hr-ft$^2$ |
| $K_Ga$ | 18.18 lb-mol/hr-ft$^3$ (atmos) |

73. Tests are made on the extraction of acetic acid from a dilute aqueous solution by means of a ketone, in a small spray tower of diameter 1·84 in. and effective height 43 in., the aqueous phase being run into the top of the tower. The ketone enters free from acid at a rate

of 16 ft³/hr-ft², and leaves with an acid concentration of 0·0235 lb-mol/ft³. The concentration in the aqueous phase falls from 0·074 to 0·051 lb-mol/ft³.

Calculate the overall extraction coefficient based on the concentrations in the ketone phase, and the height of the corresponding overall transfer unit.

The equilibrium conditions are expressed by the concentration of acid in the ketone phase being 0·548 times that in the aqueous phase.

74. A laboratory examination is made of the extraction of acetic acid from dilute aqueous solution, by means of methyl iso-butyl ketone, using a spray tower of 1·84 in. diameter and 42·5 in. high. The aqueous liquor is run into the top of the tower and the ketone enters at the bottom.

The ketone enters at a rate of 26·2 ft³/hr-ft² of tower cross-section, containing no acetic acid, and leaves with a concentration of 0·0131 lb-mol/ft³. The aqueous phase flows at a rate of 15·8 ft³/hr-ft² of tower cross-section and enters containing 0·0424 lb-mol-acid/ft³.

Calculate the overall extraction coefficient based on the driving force in the ketone phase. What is the corresponding value of the overall H.T.U., based on the ketone phase?

Using units of lb-mol/ft³, the equilibrium relationship under these conditions may be taken as:—

Concentration of acid in the ketone phase equals 0·548 times the concentration in the aqueous phase.

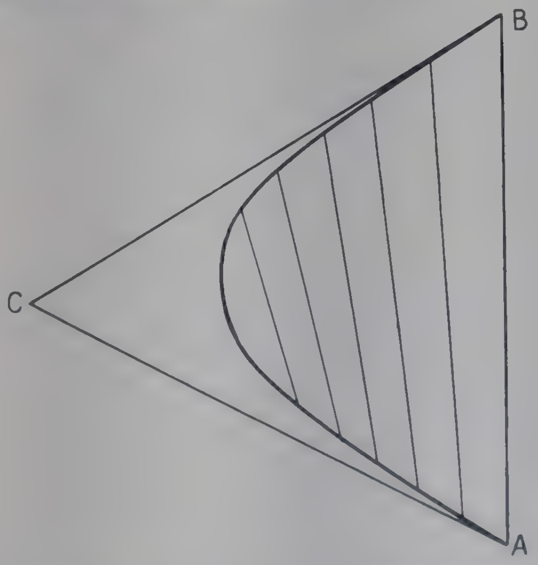
75. Propionic acid is extracted with water, from a dilute solution in benzene by bubbling the benzene phase into the bottom of a tower to which water is fed at the top.

The tower is 4 ft high and 1½ ft² in area and the perforated plate is such that the drop volume is 0·12 cc and the velocity of rise 12 cm/sec.

From experiments in the laboratory the value of $K_W$ for forming drops is 0·9 lb-mol/hr-ft² (lb-mol/ft³) and for rising drops $K_W = 0{\cdot}5$ lb-mol/hr-ft² (lb-mol/ft³).

What do you expect to be the value of $K_Wa$ for the tower in lb-mol/hr-ft³ (lb-mol/ft³)?

76. A 50% solution of solute $C$ in solvent $A$ is extracted with a second solvent $B$ in a countercurrent multiple contact extraction unit. The weight of $B$ is 25% that of the feed solution, and the equilibrium data are as given in the diagram.



Determine the number of ideal stages required, and the weight and concentration of the first extract if the final raffinate has 15% of solute A.

77. Acetaldehyde (5%) is in solution in toluene and is to be extracted with water in a five stage co-current unit. If 25 lb of water are used per 100 lb of feed, find the amount of acetaldehyde extracted and the final concentration.

If $Y$ = lb acetaldehyde/lb water, and $X$ = lb acetaldehyde/lb toluene then $Y_e = 2{\cdot}20X$ represents the equilibrium relation.

78. If a drop is formed in an immiscible liquid, show that the average surface available during formation of the drop is $\frac{12}{5}\pi r^2$, where $r$ is the radius of the drop, and that the average time of exposure of the interface is $\frac{3}{5}t_f$, where $t_f$ is the time of formation of the drop.

79. Acetic acid is being extracted from an aqueous solution in benzene in a small laboratory packed column. One set of data is given as below. Find the transfer coefficient $K_Ba$, and $\mathbf{H}_{OB}$.

Effective volume of column = 0·2214 ft³
Aqueous flow rate 29·25 ft/hr.
Benzene flow rate 11·9 ft/hr as dispersed phase.
Acid in is 0·7385 Normal and acid out is 0·7125 Normal.
Benzene in is 0·0002689 Normal and out is 0·0008276 Normal.
For these concentrations $C_B/C_W$ at equilibrium is 0·000252.

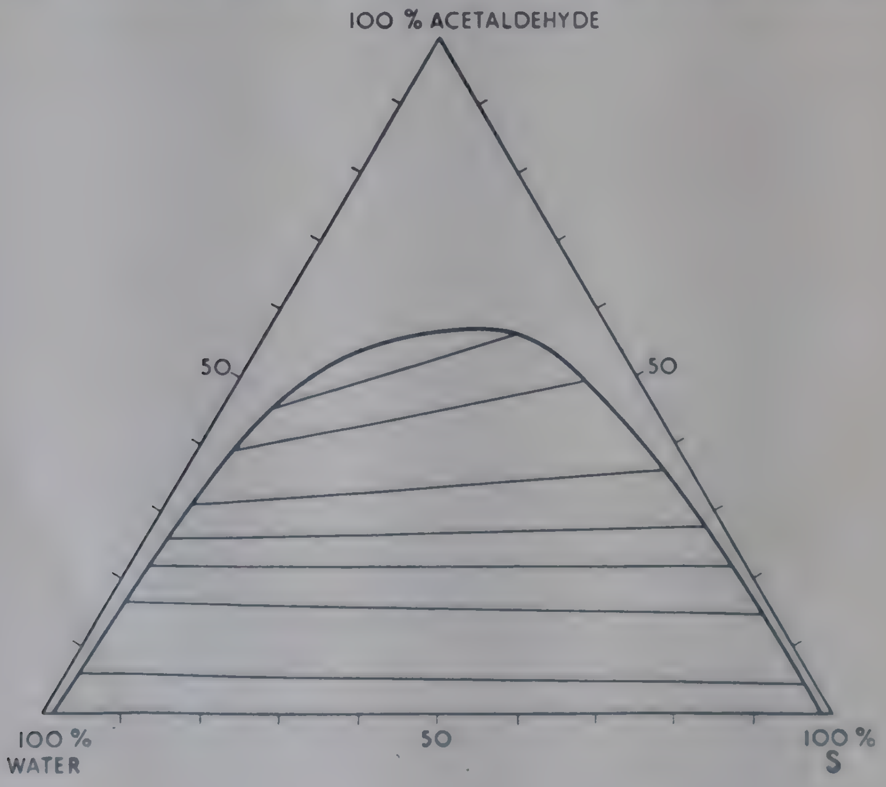
80. It is required to design a spray tower for the extraction of benzoic acid from solution in benzene.

Some experiments have been made on the rate of extraction of benzoic acid from a dilute solution in benzene to water, in which the benzene phase was bubbled into the base of a 1 in. diameter column and the water fed to the top of the column. Arrangements were made to measure the rate of mass transfer during the formation of the bubbles in the water phase and during the rise of the bubbles up the column. For conditions where the drop volume was 0·12 cc, the velocity of rise 12·5 cm/sec, the value of $K_W$ for the period of drop formation was 0·89 lb-mol/hr-ft² (lb-mol/ft³), and for the period of rise 0·55 in the same units.

If these conditions of drop formation and rise are reproduced in a spray tower of 6 ft height and 0·4 ft² cross-sectional area, what value would you expect for the transfer coefficient, $K_Wa$, in lb-mol/hr-ft³ (lb-mol/ft³), where $a$ represents the interfacial area in ft² per unit volume of the column? The benzene phase enters at 38 cc per sec.

81. It is proposed to reduce the concentration of acetaldehyde in aqueous solution from 50% to 5% by weight, by extraction with solvent $S$ at 20°C. If a countercurrent multiple contact process is adopted and 200 lb/hr of the solution are treated with an equal quantity of solvent, determine the number of theoretical stages required and the weight and concentration of the extract from the first stage.

The equilibrium relationship for this system at 20°C is given on the diagram.



82. A double effect forward-feed evaporator is required to give a product which contains 50·0% by weight of solids. Each effect has 100 ft$^2$ of heating surface and the heat transfer coefficients are known to be 500 and 300 B.Th.U./ft$^2$-hr-°F in the first and second effects respectively. Dry and saturated steam is available at 40 lb/in.$^2$-G and the condenser operates under a vacuum of 26 in.-Hg. The concentrated solution exhibits a boiling point rise of 5°F.

What is the maximum feed rate if the feed contains 10% solids and is at a temperature of 100°F?

Assume a latent heat of 1000 B.Th.U./lb and a specific heat of 1·0 under all conditions.

83. You are required to consider proposals for concentrating fruit juice by evaporation. It is proposed to use a falling film evaporator and to incorporate a heat pump cycle with ammonia as the medium. The ammonia in vapour form enters the evaporator at 103°F and the water is evaporated from the juices at 58°F. The ammonia in the vapour-liquid mixture enters the condenser at 41°F and the vapour then passes to the compressor. It is estimated that the work in compressing the ammonia will be 65·9 B.Th.U./lb of ammonia and that 2·28 lb of ammonia are cycled per pound of water evaporated. The following proposals are made for driving the compressor:

(*a*) To use a diesel engine drive taking ½ lb of fuel per B.H.P.-hr, the calorific value of the fuel being 18,000 B.Th.U./lb, and the cost 2d per lb.

(*b*) To pass steam, costing 1d. for 10 lb, through a turbine which operates at 70% isentropic efficiency, between 100 and 14·7 lb per in.$^2$ abs.

Explain by means of a diagram how this plant will work, including all necessary major items of equipment. Which method would you suggest for driving the compressor?

84. A single effect evaporator is used to concentrate 55,000 lb per hour of a solution from 10 to 50% of solids. Steam is available at 15 lb/in.$^2$ gauge and evaporation takes place at 26 in. of mercury vacuum.

If the overall heat transfer coefficient is 500 lb-cal/ft$^2$-hr-°C, calculate the heating surface required and the amount of steam used if the feed to the evaporator is at 21°C and the condensate leaves the heating space at 79·5°C.

Specific heat of 10% solution 0·9
Specific heat of 50% solution 0·75.

85. A solution containing 10% of caustic soda has to be concentrated to a 35% solution at the rate of 400,000 lb per day during a year of 300 working days. A suitable single effect evaporator for this purpose (neglecting condensing plant) costs £1600 and for a multiple effect evaporator the cost may be taken as £1600*N*, where *N* is the number of effects.

Boiler steam can be purchased at 2s. per 1000 lb and the vapour produced may be assumed to be 0·85*N* lb per lb of boiler steam. Assuming that interest on capital, depreciation, and other fixed charges amount to 45% of the capital involved per annum, and that the cost of labour is constant and independent of the number of effects employed, determine the number of effects which, on the data supplied, will give the maximum economy.

86. Saturated steam is given off from an evaporator at atmospheric pressure and is being compressed by means of saturated steam at 150 lb per in.$^2$ (gauge) in a steam jet to a pressure of 5 lb per in.$^2$ (gauge). If one pound of the high pressure steam compresses 1·6 lb of the evaporated atmospheric steam, comment on the efficiency of the compressor.

87. A single effect evaporator operates at 26 in. Hg vacuum. What will be the heating surface necessary to concentrate 10,000 lb/hr of 10% caustic soda to 41%, assuming a value of *U* of 225 lb-cal/ft$^2$-hr-°C, using steam at 116°C? The heating surface is 4 ft below the liquid level.

Boiling point rise of solution = 30°C.
Feed temp. = 18°C.
Specific heat of the feed = 0·95.
Specific heat of the product = 0·78
Specific gravity of boiling liquid = 1·39.

88. Distilled water is produced from sea-water by evaporation in a single effect evaporator working on the vapour compression system. The vapour produced is compressed by a mechanical compressor of 50% efficiency, and then returned to the calendria of the evaporator Extra steam, dry and saturated at 80 lb/in.$^2$ gauge, is bled into the steam space through a throttling valve. The distilled water is withdrawn as condensate from the steam space. Half the sea-water is evaporated in the plant. The energy supplied in addition to that necessary to compress the vapour may be assumed to appear as superheat in the vapour.

Using the data given below, calculate the quantity of extra steam required in pounds per hour.

| | |
|---|---|
| Pounds of distillate produced per hour | 1000 |
| Pressure in vapour space | atmospheric |
| Temperature difference from steam to liquor | 8°C |
| Boiling-point rise of sea-water | 1·1°C |
| Specific heat of sea-water | 1·0 |

Sea-water enters the evaporator at 71°C from an external heater.

89. It is claimed that a jet booster requires 500 lb/hr of dry and saturated steam at100 lb/in.$^2$ absolute to compress 1000 lb/hr of dry and saturated vapour from 0·5 lb/in.$^2$ to 2·0 lb/in.$^2$ absolute. Show whether this claim is reasonable.

90. A forward feed double effect evaporator, having 100 ft$^2$ of heating surface in each effect, is used to concentrate 3200 lb/hr of caustic soda solution from 10% to 50% by weight. During a particular run, when the feed is at 135°F, the pressures in the two calandrias are 40 and 11·5 lb/in.$^2$ gauge respectively, while the condenser operates under a vacuum of 26 in. of mercury. Under these conditions, find

(*a*) the load on the condenser;

(*b*) the steam economy;

(*c*) the overall heat transfer coefficient in each effect.

Would there have been any advantages in using backward feed in this case?

*Data.* Assume negligible heat losses to the surroundings.

Physical properties of caustic soda solutions—

| *per cent solids by weight* | *B.Pt rise* | *Sp. ht.* | *Heat of dilution* |
|---|---|---|---|
| 10 | 3°F | 0·92 | 0 B.Th.U./lb |
| 20 | 11°F | 0·89 | 1 B.Th.U./lb |
| 30 | 27°F | 0·87 | 4 B.Th.U./lb |
| 50 | 75°F | 0·77 | 95 B.Th.U./lb |

91. A 12% glycerol-water mixture is produced as a secondary product in a continuous process plant and flows from the reactor at 650 lb/in.$^2$ and at a temperature of 250°C. Suggest with preliminary calculations, a method of concentration to 75% glycerol, in a factory which has no low-pressure steam available.

92. A forward feed, double effect, standard vertical evaporator, with equal heating areas in each effect, is fed with 40,000 lb/hr of a liquor of specific heat 1·0, and with no boiling-point rise, so that half of the feed liquor is evaporated. The overall heat transfer coefficient in the second effect is three-quarters of that in the first. Steam is fed in at 120°C and the boiling-point in the second effect is 100°C. The feed is heated by external heater to the boiling-point in the first effect.

It is decided to bleed off 2000 lb/hr of vapour from the vapour line to the second effect for use in another process. If the feed is still heated to the boiling-point of the first effect by external means, what will be the change in steam consumption of the evaporator unit?

For the purposes of calculation, the latent heat of the vapours and of the live steam may be taken as 533 lb-cal/lb.

93. A liquor containing 15% solids is concentrated to 55% solids in a double effect evaporator, operating under a vacuum in the second effect of 24·70 in. of Hg. No crystals are formed. The feed is 20,000 lb/hr at a temperature of 215°F with a specific heat of 0·9 B.Th.U./lb-°F. The boiling point rise of the concentrated liquor is 11°F and the steam fed to the first effect is 20 lb/in.$^2$-G.

The overall heat transfer coefficients in the first and second effects are 330 and 110 B.Th.U./hr-ft$^2$-°F, respectively. If the heat transfer area is to be the same in each effect, determine its value.

94. Liquor containing 5% solids is fed at 70°C to a four effect evaporator. Forward feed is used to give a product containing 28·5% solids. Do the following figures indicate normal operation? If not, suggest a reason for the abnormality.

| Effects | I | II | III | IV |
|---|---|---|---|---|
| % solids entering | 5·0 | 6·6 | 9·1 | 13·1 |
| Temperature in steam chest °C | 109 | 101 | 94 | 84·5 |
| Temperature of boiling solution °C | 96·5 | 91·5 | 86·6 | 63·4 |

95. 10,000 lb/hr of a solution is concentrated from 10 to 50% solids in a triple effect evaporator using steam at 249°F and a vacuum such that the boiling point in the last effect is 125°F.

If the feed is initially at 75°F and a backward feed is used, what will be the steam consumption per hour, the temperature distribution in the system and the heat transfer area in each effect, which is to be the same throughout?

For the purposes of calculation, assume that the specific heat is 1, that there is no boiling point rise, and that the latent heat of vaporisation is constant at 1000 B.Th.U./lb over the temperature range in the system. The overall heat transfer coefficient may be taken as 450, 350 and 280 B.Th.U./hr-ft$^2$-°F in the first, second and third effects.

96. A triple effect evaporator concentrates a liquid with no appreciable elevation of boiling point. If the temperature of the steam to the first effect is 120°C, and vacuum is applied to the third effect, so that the boiling point is 52°C, what are the approximate boiling points in the three effects? The overall heat transfer coefficients may be taken as 550, 400, 200 lb-cal/hr-°C-ft$^2$ in the three effects.

97. A 3-stage evaporator is fed with 10,000 lb/hr of a liquor which is concentrated from 10–40% solids. The heat transfer coefficients may be taken as 550–450–300 B.Th.U./hr-ft$^2$-°F.

Calculate the steam at 10 lb/in.$^2$-G. required per hour and the temperature distribution in the three effects:—

(*a*) If the feed is at 70°F.

(*b*) If the feed is at 180°F.

Forward feed is used in each instance and the values of $U$ may be considered the same for the two systems. The boiling point in the third effect is 125°F; the liquor has no boiling point rise.

98. An evaporator operating on the thermo-recompression principle employs a steam ejector to maintain atmospheric pressure over the boiling liquid. This ejector uses 300 lb/hr of steam at 80 lb/in.$^2$-G. superheated 100°C, and produces a pressure in the steam chest of 15 lb/in.$^2$-G. A condenser removes surplus vapour from the atmospheric pressure line.

What is the capacity and the economy of the system?

How could the economy be improved?

*Data.* Properties of the ejector:—

| | |
|---|---|
| Nozzle efficiency | 0·95 |
| Efficiency of momentum transfer | 0·80 |
| Efficiency of compression | 0·90 |

The feed enters the evaporator at 20°C and thick liquor is withdrawn at a rate of 200 lb/hr. The concentrated liquor exhibits a boiling point rise of 10°C. The plant is lagged sufficiently to render negligible heat losses to the surroundings.

99. A single effect evaporator is used to concentrate 610 lb/hr of a 10% caustic soda liquor to 30%. The unit employs forced circulation in which the liquor is pumped through the vertical tubes of the calandria which are $1\frac{1}{4}$ in. o.d. by 1·12 in. i.d., and 4 ft long.

Steam is supplied at 250°F, dry and saturated, and the boiling point rise of the 30% solution is 27°F. If the overall heat transfer coefficient is 310 B.Th.U./ft$^2$-hr-°F, how many tubes do you suggest should be used, and what material of construction would you advise for the evaporator? The latent heat of vaporization under these conditions is 975 B.Th.U./lb.

100. A steam jet booster compresses 800 lb/hr of dry and saturated vapour from 29 in. vacuum to 26 in. vacuum. H.P steam consumption is 400 lb/hr at 85 lb/in.$^2$ gauge. (*a*) What must be the condition of the H.P. steam for the booster discharge to be superheated 20°C? (*b*) What is the overall efficiency of the booster if the compression efficiency is assumed equal to 1?

101. A triple-effect backward-feed evaporator concentrates 40,000 lb/hr of liquor from 10% to 50% solids. Steam at 40 lb/in.$^2$-G is available and the condenser operates under a vacuum of 26 in. Hg. Find the area required in each effect (areas to be equal) and the economy of the unit.

Assume that the specific heat is 1 at all concentrations and that there is no boiling point rise. The overall heat transfer coefficients are 400, 350 and 300 B.Th.U./ft$^2$-hr-°F respectively in the three effects. The feed enters the third effect at 80°F.

102. A double effect evaporator of the climbing film type is connected so that the feed passes through two preheaters, one heated by vapour from the first effect and the other by vapours from the second effect. The condensate from the first effect is passed into the steam space of the second. The temperature of the feed is initially at 15°C, after the first heater 75°C and after the second heater 110°C. The vapour temperature in the last effect is 125°C and in the second 100°C. The feed is 1000 Kg/hr and the steam is dry and saturated at 140°C. Find the economy of the unit if the evaporation rate is 500 Kg/hr.

103. A triple effect evaporator is fed with 40,000 lb/hr of a liquor containing 15% solids. The concentration in the last effect, which operates under a vacuum of 26 in. of mercury, is 60% solids.

If the overall heat transfer coefficients are 450, 350 and 200 lb-cal/hr-ft$^2$-°C respectively and the steam is fed at 115°C to the first effect, determine the temperature distribution and the area of heating surface required in each effect. The calendrias are to be identical. What is the economy and what is the heat load on the condenser?

Feed temperature 21°C
Specific heat of all liquors is unity

If the unit is run as a backward feed system, the coefficients are 400, 350 and 275. Under these conditions, determine the new temperatures, heat economy and heating surface required.

104. A wet solid is dried from 35% to 10% moisture, under constant drying conditions in 5 hours. If the equilibrium moisture content is 4% and the critical moisture content is 14%, how long will it take to dry to 6% moisture under the same conditions?

105. Strips of material $\frac{3}{8}$ in. thick are dried under constant drying conditions from 28 to 13% moisture in 7 hours. If the equilibrium moisture content is 7%, find the time taken to dry 2·5 in. planks from 22 to 10% moisture under the same conditions, assuming no loss from the edges. All moistures are given on the wet basis.

The relation between $E$, the ratio of the average free moisture content at time $t$ to the initial free moisture content, and the parameter $r$ is given by

| $E$ | 1 | 0·64 | 0·49 | 0·38 | 0·295 | 0·22 | 0·14 |
|---|---|---|---|---|---|---|---|
| $r$ | 0 | 0·1 | 0·2 | 0·3 | 0·5 | 0·6 | 0·7 |

Note that $r = \frac{4kt}{l^2}$, where $k$ is a constant, $t$ the time in hours and $l$ the thickness of the sheet of material in inches.

106. A granular material containing 40% moisture is fed to a countercurrent rotary dryer at a temperature of 20°C and is withdrawn at 35°C containing 5% moisture. The air supplied, which contains 0·006 lb water vapour per lb of dry air, enters at 110°C and leaves at 38°C. The dryer handles 1000 lb wet stock per hour.

Assuming that radiation losses amount to 5 lb-cal per lb of dry air used, determine the weight of dry air supplied to the dryer per minute and the humidity of the air leaving it.

| | |
|---|---|
| Latent heat of water vapour at 20°C | 585 lb-cal/lb |
| Specific heat of dried material | 0·21 |
| Specific heat of dry air | 0·238 |
| Specific heat of water vapour | 0·48 |

107. One ton (dry weight) of a non-porous solid is dried under constant drying conditions with an air velocity of 2·4 ft/sec, so that the area of surface drying is 600 ft². If the initial rate of drying is 0·24 lb/ft²-hr, how long will it take to dry the material from 0·15 to 0·205 lb of water/lb of dry solid. The critical moisture content of the material may be taken as 0·125 lb of water/lb of dry solid.

If the air velocity were increased to 13 ft/sec, what would be the anticipated saving in time if surface evaporation controls?

108. A saturated solution containing 3000 lb of potassium chloride at 90°C is cooled in an open tank to 20°C. If the specific gravity of the solution is 1·2, the solubility of potassium chloride per 100 parts of water is 53·55 at 90°C and 34·5 at 20°C, calculate—

(*a*) the capacity of the tank required, and

(*b*) the weight of crystals obtained, neglecting loss of water by evaporation.

109. What do you understand by the term "fractional crystallisation"? Explain with the aid of a diagram how the operation can be applied to a mixture of sodium chloride and sodium nitrate, given the following data. At 20°C solubility of sodium chloride is 36 parts per 100 of water and of sodium nitrate 88 parts per 100 of water. Whilst at this temperature, a saturated solution comprising both salts will contain 25 parts of sodium chloride and 59 parts of sodium nitrate per 100 parts of water. At 100°C these figures per 100 parts of water are 40, 176, and 17 and 160 respectively.

110. A material is crushed in a Blake jaw crusher and the average size of particle reduced from 2 in. to ½ in., with consumption of energy at the rate of 5 h.p. hr/ton. What will be the consumption of energy needed to crush the same material of average size 3 in. to an average size of 1 in.,

(*a*) assuming RITTINGER's law applies,

(*b*) assuming KICK's law applies?

Which of these results would you regard as being more reliable and why?

111. A crusher was used to crush a material whose compressive strength was 3300 lb/in.² The size of the feed was *minus* 2 in., *plus* 1½ in. and the power required was 5 horsepower per ton per hour. The screen analysis of the product was as follows:

| *Size of aperture* | | *Per cent of product* |
|---|---|---|
| Through | 0·25 in. | all |
| On | 0·15 in. | 26 |
| On | 0·08 in. | 18 |
| On | 0·03 in. | 23 |
| On | 0·02 in. | 8 |
| On | 0·01 in. | 17 |
| On | 0·005 in. | 3 |
| Through | 0·005 in. | 5 |

What would be the power required to crush one ton per hour of a material of compressive strength 7000 lb/in.² from a feed *minus* 1¾ in., *plus* 1½ in. to a product of average size 0·02 in.?

112. A crusher, in reducing limestone of crushing strength 10,000 lb per in. from ¼ in. diameter average size to 1/300 in. diameter average size, requires 12 horse-power. The same machine is used to crush dolomite at the same rate of output from ¼ in. diameter average size to a product which consists of 20% with an average diameter of 1/100 in., 60% with an average diameter of 1/200 in., the balance having an average diameter of 1/300 in. Estimate the horse-power required to drive the crusher, assuming that the crushing strength of the dolomite is 15,000 lb/in.$^2$ and that crushing follows RITTINGER's law.

113. If crushing rolls 40 in. diameter are set so that the crushing surfaces are 0·5 in. apart and the angle of nip is 31°, what is the maximum size of particle which should be fed to the rolls?

If the actual capacity of the machine is 12% of the theoretical, calculate the throughput in tons per hour when running at 100 revs. per minute, if the working face of the rolls is 16 in. long and the feed weighs 147 lb per ft$^3$.

114. A crushing mill reduces limestone from a mean particle size of 1¾ in. to a product,

| | |
|---|---|
| size 0·50 in. | 0·5% |
| 0·30 | 7·5 |
| 0·20 | 45·0 |
| 0·10 | 19·0 |
| 0·06 | 16·0 |
| 0·03 | 8·0 |
| 0·015 | 3·0 |
| 0·007 | 1·0 |

and in so doing requires 8 h.p. per ton of material crushed.

Calculate the power required to crush the same material at the same rate, from a feed having a mean size of 1 in. to a product with a mean size of 0·04 in.

115. A ball-mill 4 ft in diameter is being run at 50 revolutions per minute; it is found that the mill is not working satisfactorily. Would you suggest any modification in the conditions of operation?

116. 4 h.p. has to be supplied to a machine crushing material at the rate of 1 ton per hour from ½ in. cubes to a product having the following sizes:—

| | |
|---|---|
| 80% | 0·125 in. |
| 10% | 0·1 in. |
| 10% | 0·09 in. |

What would be the horse-power which would have to be supplied to this machine to crush 1 ton per hour of the same material from 0·3 in. cube to 0·08 in. cube?

117. The size analysis of a powdered material on a weight basis is represented by a straight line from 0% weight at 1 micron particle size to 100% weight at 101 microns particle size. Calculate the surface mean diameter of the particles constituting the system.

118. The equations giving the number distribution curve for a powdered material are $\frac{dn}{dd} = d$ for the size range 0 to 10 microns, and $\frac{dn}{dd} = \frac{100{,}000}{d^4}$ for the size range 10 to 100 microns. Sketch the number, surface and weight distribution curves. Calculate the surface mean diameter for the powder.

Explain briefly how the data for the construction of these curves would be obtained experimentally.

119. The fineness characteristic of a powder on a cumulative basis is represented by a straight line from the origin to 100% undersize at particle size 50 microns. If the powder is initially dispersed uniformly in a column of liquid, calculate the proportion by weight which remains in suspension at a time interval from commencement of settling such that a 40 micron particle would fall the total height of the column.

120. In a mixture of quartz, specific gravity 2·65, and galena, specific gravity 7·5, the sizes of the particles range from 0·00052 to 0·0025 cm.

On separation in a hydraulic classifier under free settling conditions three fractions are obtained, one consisting of quartz only, one a mixture of quartz and galena, and one of galena only. What are the ranges of sizes of particles of the two substances in the mixed portion?

121. It is desired to separate into two pure fractions a mixture of quartz and galena of a size range from 0·00058 to 0·00250 cm by the use of a hindered settling process. What is the minimum apparent density of the sorting bed that will give this separation? How will the viscosity of the bed affect the minimum required density?

Specific gravity of galena = 7·5. Specific gravity of quartz = 2·65

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